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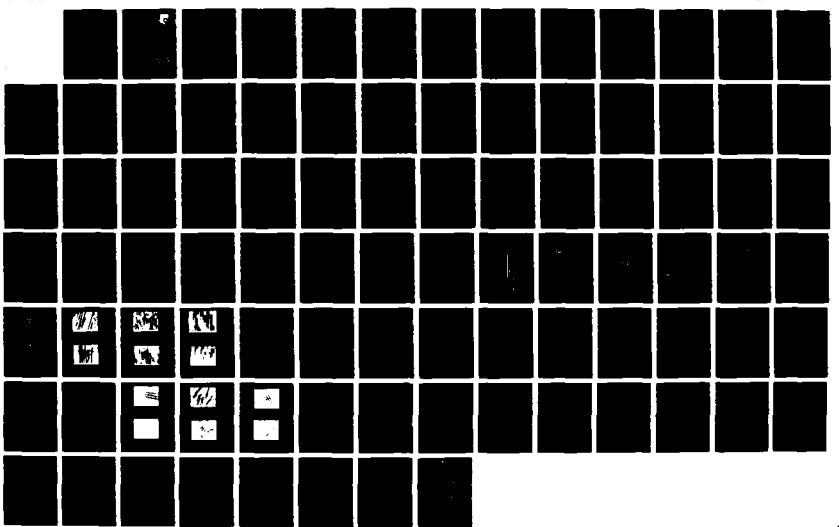

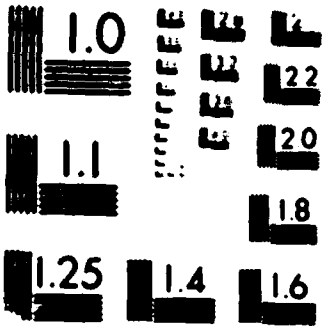

MICROCOPY RESOLUTION TEST CHART

AFWAL-TR-87-4136

AT TECHNOLOGY TRANSITION

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MARCH 1988

FINAL Report for Period March 1985 - August 1987

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This technical report has been reviewed and is approved for publication.

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(U) Formulations of two AT matrix resin candidate systems, ATB and ATS, supplied by the Air Force and formulated for improved toughness by the Contractor, were screened via a neat resin evaluation study. The most promising formulations were prepregged on graphite fiber, fabricated into composite specimens, and subjected to an extensive composite mechanical properties evaluation. For each resin system, the optimum formulation was found to display significantly improved toughness and 400 degree F use capability. The optimum formulation for each resin was utilized to prepare a 25 pound batch of graphite fiber reinforced prepreg which was divided into 4 pound samples and distributed to various airframe and missile companies for evaluation.

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FOREWORD

This final report covers the work performed under Air Force Contract F33615-84-C-5115, "AT Technology Transition," during the period March 1, 1985 to August 7, 1987. This program was conducted under the technical direction of Dr. Frederick L. Hedberg, Air Force Aeronautical Laboratories.

The contract program was carried out at Hysol Grafil Company, Pittsburg, CA. Mr. Jim Browne was Program Manager and Dr. Paul A. Steiner, the Principal Investigator. Much of the data presented in this report was made possible through the diligent efforts of John McKillen. Mr. Browne, Dr. Steiner and John McKillen are employees of Dexter Hysol Aerospace, Inc. Their efforts were made available to Hysol Grafil by Hysol for the purpose of performing this effort.

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1.0 INTRODUCTION

The development of acetylene terminated (AT) resin systems began at the Air Force Wright Aeronautical Laboratories in the early 1970's.[1] After several materials had been synthesized and initially characterized, two materials, 4,4'-bis (3-ethynlphenoxy) isopropylidenediphenol (m-ATB) and 4,4'-bis (3-ethynlphenoxy) diphenylsulfone (m-ATS) were identified as the members of that family of resins which were most likely to possess the desired uncured resin properties (low Tg or melt temperature), processing characteristics comparable to epoxy resins, high temperature composite mechanical properties, and the potential for low cost synthesis.[2,3] At a resin composition which is approximately 75% monomer and 25% oligomer, the bisphenol A backbone gives a resin which is a viscous liquid at ambient temperature while the sulfone backbone gives an amorphous low melting solid. The acetylene terminated resins display excellent pot life and can be thermally homopolymerized via free-radical addition reactions leading to a conjugated polyene without the evolution of volatile by-products. The polymer network present during the early stages of cure is pictured in Figure 1. The cured resins have high thermal and thermal oxidative stability making them suitable for long term use at 400-450°F and short term use at 550-600°F.[4] Their hydrophobic nature results in a relatively moisture insensitive cured resin with good properties retention after exposure to high humidity conditions.

State-of-the-art structural epoxies and bismaleimides have had drawbacks in the area of moisture aging and hot/wet use temperatures. This has limited the use of epoxies and bismaleimides to 180°F and 350°F, respectively, in humid environments. AT resins are not subject to these limitations and, therefore, display improved hot/wet mechanical properties at 400-450°F.[5] Unfortunately, like epoxies and bismaleimides, the AT resins exhibit a high degree of brittleness making the composite susceptible to damage by impact. The intent of this Air Force sponsored research was to toughen the m-ATB and m-ATS resins so that they may be considered for use in primary aircraft structural applications.

This program consisted of two phases with Phase 1 evaluating m-ATB and Phase 2 evaluating m-ATS. Prior to the start of each phase several formulations were developed for each of the AT resins under IR&D funding. The formulations developed were transitioned into the contract for evaluation according to the program plan in Figure 2.

2.0 TECHNICAL DISCUSSION OF APPROACH

2.1 Program Objective

The objective of this program is to develop improved graphite fiber/acetylene terminated matrix resin prepregs through the development of optimum formulations for m-ATB and m-ATS. It was desired that the room temperature mechanical properties of the composites be equivalent to present epoxy/graphite composites while maintaining a processability of the prepreg equivalent to these systems. The formulated resins should exhibit 400°F service capability after exposure to high humidity environments. The most critical property being sought after was a fracture toughness superior to present bismaleimide/graphite composites.

2.2 IR&D M-ATB and M-ATS Formulation

For the purposes of toughening the m-ATB and m-ATS resins, two basic approaches were established. The first was to attempt to decrease the crosslink density by the addition of monofunctional unsaturated modifiers which react with the AT resins and/or the addition of high glass transition temperature ductile polymers which remain in a continuous phase with the cured m-ATB or m-ATS. This reduced crosslink density renders the matrix more toughenable. The second approach was to increase the toughness by the addition of a more ductile second phase as is commonly accomplished by the addition of elastomers or tough glassy thermoplastics to expoxies. Additional reactive diluents were added when necessary to increase the tack, drape and handleability of the prepreg.

In order to maintain the use temperature of 400°F - 450°F for the formulated graphite fiber/acetylene terminated matrix resin prepreg, it was desirable that the modifiers used have sufficiently high Tg's so as not to seriously compromise the Tg of the unformulated resin. One of the attractive features of the acetylene terminated resins has been their very low moisture absorption in the cured composite structure. This property is achieved by the low polarity of the resin. To maintain this property and thus achieve the required 400°F service capability of the composite after 75% RH exposure to equilibrium moisture content will require that the polarity of these modifiers also be considered in their selection. Giving full consideration to the above constraints on the choice of modifiers for improved toughness, several m-ATB and m-ATS formulations were made prior to the planned initiation of the contract under IR&D funds. The most promising formulations identified during the neat resin screening calculations were carried on to the preparation of unidirectional XAS carbon fiber prepreg tapes.

2.3 Program Approach

The program was divided into two phases as outlined in Figure 2. Phase 1 accomplished the development of an improved graphite fiber/ATB matrix resin prepreg. Several ATB formulations using base ATB resin supplied by AFWAL/MLBC were prepared for evaluation. Task I in Phase 2, occurring sequentially, covered the development of an improved graphite fiber/ATS matrix resin prepreg. Each Phase is divided into four tasks. Task I involved characterization and evaluation of the neat formulated resins. The most promising formulations as determined from the neat resin testing were included in the Task II preparation of prepreg material. Chemical and physical properties were determined on these prepregs. In Task III fabrication, characterization and evaluation of composite test panels were performed. From the detailed test matrix in Task III, the best formulation was determined in Task IV for each acetylene terminated resin system investigated. Task IV was completed by the preparation of a single batch of graphite prepreg tape which was delivered to AFWAL/MLBC.

Details of Phase 1 of this program are discussed below. Phase 2 was identical in format, having Tasks I, II, III & IV and is, therefore, not separately discussed.

2.3.1 Task I - Neat Resin Characterization and Evaluation

By the start of Task I and no later than the end of the task, a minimum of four formulated ATB resins were developed. Major emphasis was placed on improving their toughness while maintaining their 400-450°F properties. Toughening methods, some of which were proprietary to Hysol, included the use of various thermoplastics which either formed a continuous phase with the ATB or separated into a second phase during cure. Other toughening modifiers were reactive monomers which polymerized during cure via a mechanism different than that of ATB to give IPN type entanglement. The formulatory work also took into consideration the need for self adhesion tack, the desire to have no objectionable odors/vapors, a 30 day out-time at 80°F and the ability to be cured with present equipment (375°F/100 psi max).

2.3.1.1 Uncured Resin Properties

The Tg of each of the four uncured resins was determined by Dynamic Mechanical Thermal Analysis (DMTA). Differential Scanning Calorimetry (DSC) to determine the cure exotherm range and a rheometric cure profile revealed information on the processability of each of the formulations which was of value in the development of an appropriate cure cycle and prepreg process conditions. The test matrix for these properties is given in Table 1.

2.3.1.2 Cured Neat Resin Properties

A standardized cure cycle based on prior AFWAL/MLBC data and the DSC and rheometrics obtained in Section 2.3.1.1 served to cure two different neat resin castings that are void free for each formulation. From one of the castings (approximately 4"x2.5"x0.05") DMTA specimens were cut and tested in accordance with Figure 3. The Tg and matrix flexural modulus obtained from the DMTA supplied information on how these materials behaved at elevated temperatures. An additional DMTA specimen was cut and immersed in 160°F water until equilibrium water uptake was attained, which is defined as until the incremental weight gain over a 7-day period is less than 0.04% of the original dry weight. The specimen weight change was recorded followed by a determination of the wet Tg and matrix flexural modulus. Increased moisture absorption has been related to depression of the apparent Tg of the material and, in turn, lowers the elevated-temperature properties of the graphite composite under matrix-dominated stress conditions. The decrease in matrix modulus is an indication of the expected decrease in matrix dominated composite properties. The Tg and modulus are, therefore, meaningful parameters indicating the degree of moisture effects on the matrix. Portions of the casting were also ground to a fine powder. The powders were used in a thermal gravimetric analysis (TGA) run in air and nitrogen to identify the thermal oxidative and thermal stability, respectively, of each formulation. This information indicates how these materials might behave when used in an elevated temperature environment.

From the second resin casting (approximately 12"x11"x0.125") five compact tension specimens (Figure 4) were cut as indicated in the test matrix in Table 1. G_{1c} measurements from the compact tension specimen were conducted according to ASTM E399-78a. Specimens were machined to comply with specifications and a sharp precrack was introduced while the specimen was observed under a microscope. Tests were conducted on an Instron universal test machine at a crosshead speed of 0.05 inch/minute at room temperature. G_{1c} is a measure of the mode 1 - plane - strain fracture toughness of the neat resin and it can be correlated to the toughness of the graphite composite laminate.

2.3.1.3 Neat Resin Cure Cycle Optimization

Variations in the cure cycle used in Section 2.3.1.2 were made based on DSC's from pieces of the cured casting for each of the formulations used in that section with the desire to bracket the cure cycle used. If it was found

necessary to make a change in the cure cycle the cured neat resin tests were duplicated for one to all of the AT formulations.

2.3.2 Task II - Preparation of Prepreg Material

The most promising formulations as determined from the neat resin testing of Task I and approved by the Project Scientist were selected for graphite tape preparation. Sufficient graphite prepreg tape for each formulation was prepared to accommodate the fabrication and testing required in Task III. This material was produced on a full scale unidirectional prepreg production machine.

A prepreg physical properties data sheet, typical of that used in this program, is shown in Figure 5. This data sheet was used to record each of the following tests:

- Resin content/graphite areal weight was determined from three 4x4 inch samples of the prepreg. After weighing the squares the resin matrix was dissolved with appropriate solvent(s). The fibers were separated by filtration, dried at 220±10°F for 30 minutes, and then allowed to cool to room temperature in a desiccator at which point they were weighed. From this the resin content percent and graphite area weight was calculated.

- Volatile content was determined by measuring the weight difference following a 20±5 minute exposure in an air circulating oven at 325±10°F.

- Resin flow was determined by heating a four inch square layup (0/90/90/0) sandwiched between two three-ply layers of glass fabric separated from the layup by one ply of perforated Teflon in a press at 350°F and 100 psi for 5 minutes plus the gel time.

- Gel time was determined using a Fisher-Johns melting-point apparatus at 170±2°C(338±3.6°F). The gel point was reached on a 0.25x0.25 inch piece of prepreg placed between two cover glasses when no resin movement was observed while at room temperature.

- Tack was judged sufficient if two 1 x 3 inch prepreg specimens adhered to each other and a vertical corrosion-resistant steel plate for 30 minutes while at room temperature.

- Out time at room temperature was judged as the time over which the prepreg still passed the tack test. At the same time the build-up of resin viscosity with time at room temperature was monitored by rheometrics.

2.3.3 Task III - Fabrication, Characterization, and Evaluation of Composite Test Panels

2.3.3.1 Lay-up, Bagging Procedure and Cure/Post Cure Cycle Development

Cure cycle development work for the neat resin, carried out in Task I (Section 2.3.1.3), served as the basis for the cure cycle development for the graphite/AT prepregs. To enable curing of the prepreg with a majority of the autoclaves in service, upper limits of 375°F and 100 psi were established. As a rule of thumb, the cure cycle should be as simple as possible. Ideally, the autoclave pressure should be kept constant throughout the entire cure cycle with the temperature maintained at a single value after heat-up. By having no changes in the temperature or pressure during the cure cycle, it is more likely that the cure state of the composite laminates will be identical from batch to batch.

Since the AT resins require a higher temperature than 375°F to be fully cured an unrestrained post cure at a maximum temperature of 650°F follows the autoclave cure. As before, the temperature and length of this post cure was based on the cure cycle development work of Task I. Evaluation of the cure/post cure cycles developed was accomplished by DSC on cured laminates to check for residual exotherm, DMTA to compare the laminate Tg with the optimized neat resin Tg from Section 2.3.1.2 and 0-deg short beam shear laminate testing. The cure/post cure cycle should result in $[(+30,-30)_2,90]_S$ and $(\pm 45°)_{2S}$ crack-free laminates when fabricated and examined under an optical microscope. Processability of the prepreg in a manner similar to graphite/epoxy prepreg was one of the target properties. A lay-up bagging procedure similar to the one in Figure 6 for graphite/epoxy prepreg was used.

2.3.3.2 Screening Mechanical Testing of AT Composite Laminates

Composite mechanical tests were conducted on each of the prepreg samples transitioned into this task. The test matrix (Table 2) was designed to be able to delineate candidates for subsequent more comprehensive testing with as few tests as possible. The screening tests are as follows:

Fiber volume for each fabricated laminate was calculated from the measured laminate, resin, and fiber densities. Resin density, fiber density, and laminate weight were used to calculate void content. This method is accurate only for void contents above 2% because of extreme sensitivity to small changes in fiber and/or resin density.

Thus, it provided only an initial screening for laminates at a void content level high enough to have direct effects on laminate mechanical properties. Cross sectioning, polishing, and microscopic examination were used to provide an indication of voids below the 2% level where they are unlikely to have an effect.

Changes in mechanical behavior due to moisture exposure have been observed in such matrix-dominated properties as compression strength and shear modulus. These changes in mechanical behavior are more pronounced at elevated temperatures. Because significant differences in compression properties are caused by changes in temperature and by moisture pickup in the composite laminate, the 350°F and 425°F compression strength was determined in the 0-deg direction to show the ability of the matrix to stabilize the graphite filaments. The test specimen is shown in Figure 7.

The weight change that occurred in laminate specimens immersed in 160°F water to equilibrium was monitored until the incremental weight gain over a 7-day period was less than 0.04% of the original dry weight. Each specimen weight change was recorded, as well as the weight changes of control specimens. It has been found that use of control specimens yields more accurate weight-gain measurements.

Interlaminar shear depends entirely on the shear strength between the fiber and matrix. This property will fall off dramatically at higher temperatures (above matrix Tg) and is, therefore, a good screening test to assess the high temperature properties of the AT formulation. This specimen is shown in Figure 8.

Considering the fact that the objective of this program is to develop an acetylene terminated matrix resin with improved toughness, it seems only natural that mechanical testing to measure this composite property would occur at this point. The mode I critical strain energy release rate, G_{1c}, was measured at room temperature using the uniform double cantilever beam test (Figure 9).

2.3.3.3 Detailed Mechanical Testing of AT Composite Laminates

Based on the results of Section 2.3.3.2, two of the AT prepregs were subjected to detailed mechanical testing as shown in the test matrix in Table 2. All mechanical property testing was performed using test machines complying with ASTM E4. Specimens tested at room temperature were tested at 75±10°F. Dry test specimens and environmentally exposed specimens were held for 10±3

minutes and 2±1 minute, respectively, prior to initiating test load. These tests serve to fully characterize the AT prepregs with respect to room and high temperature dry and wet properties and toughness.

The void content, fiber volume and density were measured just as in Section 2.3.3.2 for each of the laminates. Tensile strength and modulus were determined in the 0-deg direction to define fiber-dominant properties. The test specimen used is shown in Figure 10. Present ultimate tensile strengths are limited by the severity and frequency of fiber flaws. The matrix must be able to redistribute the load under conditions of high shear caused by isolated fiber breaks. The inability of the matrix to redistribute the load is magnified at lower temperatures and, therefore, this test was carried out at -67°F and room temperature.

Measurement of the 0-degree dry-compression strength at room temperature, 350°F and 450°F helps to monitor the behavior of the matrix with increasing temperature and serves as a comparison for the hot/wet compression strength measured in Section 2.3.3.2. 0-deg interlaminar shear (Figure 8) and flexure (Figure 11) are both relatively easy specimens to prepare. As described in Section 2.3.3.2 the 0-deg interlaminar shear measures the shear strength between the fiber and matrix. Therefore, test results should point out any weakness that may exist in this interfacial region. Poor interfacial bonding is usually magnified under wet conditions so room temperature, 350°F and 425°F wet tests were conducted. Two types of shear tests were performed, the 0-deg just described and a ±45-deg in-plane shear test. The wet in-plane shear modulus was measured using a tensile coupon (Figure 10) with the fibers oriented ±45-deg to the length of the specimen.

When a specimen is tested in flexure, one face is under compression while the other is in tension so that the flexure strength and modulus are a good measure of overall composite properties. Since it is an easy specimen to make, the flexure test is a good test to measure the composite properties over a wide range of temperatures. The 0-degree flexure strength and modulus was measured at room temperature and elevated temperatures both wet and dry.

90-degree flexure (Figure 11), a matrix dominated property in which one of the faces of the specimen places the matrix in tension was tested at room temperature.

A final test which was used as a measure of the toughness of a composite was the edge delamination tension test (Figure 12) which was used to measure the interlaminar fracture toughness G_{1c} for the composite. The specimen was tested at room temperature dry to give the edge delamination strength.

Composite specimens with a $(0,+45,-45,0)_S$ orientation were subjected to 25 hours at 350°F followed by 264 hours at 275°F. At the end of this time a polished edge of each of the laminates was examined under microscope for microcracks. In the thermal spike test, laminates having the same lay-up as those above were immersed in 160°F water. Every 48 hours the laminates were subjected to a thermal spike at 350°F for 10 minutes and the percent weight gain over the control laminates (not spiked) was measured.

2.3.4 Preparation of Additional Prepreg Material

The best formulation was determined from the testing described in Task III with approval by the Project Scientist. A graphite prepreg tape sample of this formulation was prepared from 25 pounds of the unformulated acetylene terminated resin supplied by AFWAL/MLBC. This graphite prepreg was characterized with respect to the conventional prepreg characterization tests (Figure 5) and on completion of these tests the prepreg was delivered to AFWAL/MLBC.

3.0 SUMMARY

The goal of this program has been the development of toughened acetylene terminated resins which maintain 400-425°F service under wet conditions. The neat resin and composite mechanical properties of unmodified m-ATB and m-ATS presented in the report illustrate that m-ATS possesses superior elevated temperature properties to m-ATB. The sulfone group apparently gives this resin a higher glass transition temperature than the m-ATB and this results in the improved Young's modulus retention at elevated temperatures. As would be expected by their chemical structures (Figure 1), the more polar m-ATS had greater moisture absorption than m-ATB, but since its dry Tg was almost 70°C higher than that of m-ATB, its wet Young's modulus still surpasses that of m-ATB.

Development of the formulated m-ATB (AF-4 and AF-8) and m-ATS (AF-11 and AF-20) resins was accomplished on IR&D funding. While these formulations were not meant to be optimum formulations, they do represent the first attempts to formulate and toughen m-ATB and m-ATS. Over 20 formulations were originally developed. Those that appear in detail in the report were selected by the AFWAL project moniter to have the best overall balance of toughness and high temperature properties. From the neat resin and composite results of AF-4 and AF-8 formulations, it may be concluded that dramatic improvements have been made in the fracture toughness of m-ATB with little sacrifice in its high temperature properties. Neat resin G_{1C}'s for AF-4 and AF-8 show increases of 370% and 225%; respectively, while the composite G_{1C}'s obtained from the double cantilever beam test gave improvements of 91% and 64% respectively.

A good measure of the potential elevated temperature mechanical properties can be obtained from the dynamic mechanical thermal analysis (DMTA) modulus vs. temperature profile (Table 4). This data would indicate that m-ATB and the two formulations have roughly equivalent properties up to 350°F, but that between 350°F and 450°F the modulus of the formulations falls off more dramatically than does the m-ATB. At 450°F AF-8 retains a greater percentage of its room temperature modulus than does the AF-4. While formulations AF-4 and AF-8 exhibit more than double the equilibrium moisture weight gain of m-ATB at approximately 1.4% each, this value is extremely low when compared to resins such as epoxies and bismaleimides. The wet DMTA modulus vs. temperature profiles (Table 5) still indicate that m-ATB has the best retention of ambient temperature modulus at 425°F, but AF-4 and AF-8 are not that much lower.

Composite 0° flexure, 0° interlaminar shear, and 0° compression when tested at a variety of temperatures is a good measure of the overall composite mechanical properties. According to our neat resin results, the m-ATB composites should have had better elevated temperature performance than either AF-4 or AF-8 (Table 10.) The poor m-ATB composite properties could be due to the poor carbon fiber/resin interface which is illustrated by the degree of fiber pullout and almost no resin adhering to the fibers in the SEM photomicrograph of Figure 13. The inferior interface was especially detrimental under wet conditions where the water could potentially wick along the interface.

The improved carbon fiber/resin interfaces in the AF-4 and AF-8 composites (Figure 14) were responsible for the improved composite performance. While improved, the AF-4 and AF-8 interlaminar shear strengths were still not as high as those typically observed with epoxies or BMI's. Further strengthening of the interface would likely translate into even further improved composite properties. Resins AF-4 and AF-8 showed good retention of composite mechanical properties at 450°F/dry with AF-8 exhibiting slightly higher properties than AF-4. For example, 0° Flexure strength retention at 450°F/dry was 62% of ambient for AF-8 and 46% for AF-4. With their low equilibrium moisture absorptions, AF-4 and AF-8 exhibited good retention of wet composite mechanical properties at elevated temperatures. As with the dry properties, AF-8's wet elevated temperature performance was superior to AF-4 retaining, for example, 44% of its ambient temperature dry flexure at 425°F versus 32% for AF-4.

Since m-ATS is a low melting solid, its formulation presented some problems not encountered with the m-ATB. When comparing the glass transition temperatures and modulus versus temperature profiles, m-ATS and its formulations AF-11 and AF-20 gave significantly better high temperature properties from m-ATB, AF-4 and AF-8. The improved high temperature properties of AF-11 and AF-20 were accompanied by neat resin G_{1c}'s comparable to AF-4 and AF-8. These G_{1c}'s were 220% and 300% greater than that of m-ATS for AF-11 and AF-20, respectively. The double cantilever beam test specimens all showed a considerable degree of fiber bridging which resulted in artificially high values. However, if we assume roughly equivalent levels of fiber bridging then, the composite G_{1c}'s for AF-11 and AF-20 are 43% and 32% greater than that for m-ATS.

Examination of the neat resin glass transition temperatures and DMTA Young's modulus versus temperature profiles for AF-11 and AF-20 demonstrates that these formulations have significantly better high temperature properties than the m-ATB formulations. This was no surprise since m-ATS had much better high temperature properties than m-ATB. The excellent high temperature properties of m-ATS were largely preserved in the formulations. AF-11 and AF-20 retained 66% and 70%, respectively, of their room temperature modulus at 450°F as compared to 68% for the m-ATS. At approximately 2% the equilibrium moisture absorptions of the m-ATS formulations were higher than those of the m-ATB formulations. Since the dry elevated temperature properties of AF-11 and AF-20 were superior to AF-4 and AF-8, their wet modulus versus temperature profiles (Table 8) still exhibit higher retention of their ambient temperature modulus in spite of the greater moisture absorptions. AF-20 had better high temperature wet properties than AF-11.

Considering the significant improvements seen in the neat resin mechanical properties of the m-ATS formulations relative to the m-ATB formulations, the composite mechanical properties for AF-11 and AF-20 were baffling. Part of the problem may be traced back to the carbon fiber/resin interface. Comparison of the SEM photomicrographs in Figure 13 indicates that with more resin adhering to the fibers and less fiber pullout m-ATS had a stronger interface than m-ATB. However, the formulated m-ATS resins, AF-11 and AF-20, failed to show any further improvements in the interface, whereas AF-4 and AF-11 exhibited dramatic improvements in the m-ATB interface. As a result, AF-4 and AF-8 exhibited better carbon fiber/resin interfaces than AF-11 and AF-20. Of the two m-ATS formulations, AF-20 showed greater high temperature composite mechanical

properties and in spite of the interface problems, they were comparable to those of the m-ATB formulations. This is especially true when you consider that this resin had a neat resin G_{1c} of 0.8 in-lbs/in^2.

Development of AF-4, AF-8 and AF-11 and AF-20 represented the first attempts to formulate and toughen m-ATB and m-ATS. As such, the formulations have demonstrated composite mechanical properties superior to present state-of-the-art toughened bismaleimides achieving useful mechanical properties up to 425^{0}F under wet conditions. The weak carbon fiber/resin interfaces present in m-ATB and m-ATS have been strengthened through formulation, but more research needs to be focused on this area to allow these resins to realize their fullest potential as composite matrix resins. As more experience is gained in working with these novel addition cured thermosets, their composite mechanical properties should improve and appear even more favorable compared with the alternative resins presently available.

3.1 IR&D Interface Investigations

The SEM photomicrographs in Figure 13 show the tensile failure surfaces for unmodified m-ATS/Hysol Grafil XAS carbon fiber and m-ATB/hysol Grafil XAS carbon fiber 0 degree flexure specimens. Both resins have a poor interface with substantial fiber pullout and little resin adhering to the fibers. The m-ATB appears to be slightly worse than m-ATS as expected from its lower polarity. Figures 14 and 15 show the SEM photomicrographs of the tensile failure surfaces for AF-4, AF-8, AF-11 and AF-20. All four formulations show a dramatic improvement in interface over the unmodified resins with less fiber pullout and increased amounts of resin adhering to the fiber. The improvement in the m-ATB formulations AF-4 and AF-8 over the unmodified m-ATB appears to be greater than the improvement of AF-11 and AF-20 over the unmodified m-ATS. The more modest interface improvement of AF-11 and AF-20 over m-ATS could account for the less than expected improvement in elevated temperature composite properties. Although the AF-20 composite properties are comparable to AF-4 and AF-8, the neat resin data when considered independently of interface suggest that the AF-20 composite properties should be substantially better.

4.0 TECHNICAL DISCUSSION

A total of nine m-ATB and eleven m-ATS formulations, as well as several modifications of formulations, were developed under IR&D funding. Each of these 20 resins was evaluated for processability and cured neat resin properties by the methods described in Task I (Section 2.3.1). The neat resin data for these formulations is tabulated in Tables 3-8. Two m-ATB formulations, AF-4 and AF-8, and two m-ATS formulations, AF-11 and AF-20, were selected in consultation with AFWAL/MLBC for the preparation of prepreg material (Task II, Section 2.3.2) and the evaluation of composite test panels (Task III, Section 2.3.3). A detailed technical discussion of the neat resin, prepreg, and composite properties of these four formulations follows. These properties are contrasted and compared with unmodified m-ATB and m-ATS.

4.1 Task 1: Resin Characterization and Evaluation

4.1.1 Uncured Neat Resin Properties

4.1.1.1 Viscosity Profile During Cure

The low neat resin viscosity for m-ATB (Figure 16) and for m-ATS (Figure 17) results in difficulties during the production of prepreg and excessive flow during autoclave cure. The formulated m-ATB resins AF-4 (Figure 18) and AF-8 (Figure 19), as well as the formulated m-ATS resins AF-11 (Figure 20) and AF-20 (Figure 21), have minimum viscosities in the range that would be considered optimum for a prepreg resin. The minimum viscosities are tabulated in Tables 3 and 6 along with other neat resin properties.

4.1.1.2 DSC

Figures 22, 23 and 24 give the DSC's for m-ATB, AF-4 and AF-8. The values are tabulated in Table 3. Figures 25, 26 and 27 give the DSC's for m-ATS, AF-11, and AF-20. The values are tabulated in Table 6. The uncured DSC's of the formulations are similar to their respective unmodified controls, m-ATB and m-ATS and are therefore capable of being cured under identical conditions.

4.1.1.3 Uncured Tg and Handling Properties

The uncured Tg's for m-ATB and its formulations AF-4 and AF-8 are given in Table 3. Table 6 contains the m-ATS, AF-11, and AF-20 uncured Tg data. The Tg's of all four formulations AF-4, AF-8, AF-11, and AF-20 are lower than their respective controls m-ATB and m-ATS. All of the formulation Tg's are typical of what would be expected for a prepreg that possesses good tack, drape, and handleability at ambient temperature. All of the formulations have sufficient tack for a prepreg resin. Note that with an uncured Tg above room temperature, the m-ATS control gives a board-like prepreg with no tack.

4.1.2 Cured Neat Resin Properties

4.1.2.1 SEM Examination

The SEM photomicrographs of the cured neat resin formulations and their controls, m-ATB and m-ATS are shown in Figures 28, 29 and 30. Both the m-ATB and the m-ATS controls give a smooth fracture surface which is typical for brittle resins. All four formulations AF-4, AF-8, AF-11, and AF-20 exhibit a rough fracture surface typical of toughened resins. The AF-8, AF-11, and AF-20 formulations have a one phase morphology while in AF-4 there is a distinct second phase consisting of particles less than one micron in diameter. The particles appear to be well bonded to the continuous phase with many of them actually sheared in half without debonding from the surrounding material.

4.1.2.2 DMTA Analysis: Tg and Modulus

Figures 31 through 36 give the dynamic mechanical thermal analysis of m-ATB, m-ATS, and the four formulations. The AF-4 and AF-8 show the major transition in the 260°C to 270°C range which is comparable to the unmodified m-ATB (Tg of 260°C) while the AF-11 and AF-20 show the major transition in the 300 to 310°C range, comparable to the unmodified m-ATS (Tg of 320°C). The presence of second phase modifiers causes AF-4 to have a minor Tg of about 194°C.

Tables 4 and 7 tabulate the dry Young's modulus versus temperature for the m-ATB and m-ATS formulations, respectively. AF-8 has a slightly better retention of modulus at high temperatures than AF-4 while both have a somewhat lower retention than the unmodified m-ATB. The modulus retention of AF-11 and AF-20 are comparable and both are slightly better than unmodified m-ATS.

4.1.2.3 DMTA Analysis: Equilibrium Moisture Gain and Wet Tg

Figures 37 through 39 give the wet DMTA's for m-ATB and its formulations while Figures 40 through 42 give the wet DMTA's for m-ATS and its formulations. The moisture absorption values at equilibrium are listed in Table 3 for the m-ATB formulations and Table 6 for the m-ATS formulations. The unmodified m-ATB had the lowest moisture absorption (0.6%) while the more polar m-ATS followed with 1.09%. Note that although the moisture absorption of unmodified m-ATS was 82% greater than that for m-ATB, the AF-20 modification of m-ATS had only a 30% greater moisture absorption than the AF-4 modification of m-ATB. All four formulations have what would be considered extremely low moisture absorptions for a matrix resin (i.e. epoxy, BMI).

The wet Tg results are listed in Table 3 for the m-ATB formulations and in Table 6 for the m-ATS formulations. The wet modulus versus temperature results are listed in Tables 5 and 8 for the m-ATB and m-ATS formulations, respectively. As anticipated, there is a trend toward greater moisture absorption resulting in lower wet Tg's and lower wet modulus at elevated temperatures. The unmodified m-ATB wet Tg and modulus are close to the dry data as would be expected from the very low moisture absorption. The unmodified m-ATS also had a high wet Tg and a retention of wet modulus comparable to its retention of dry modulus. The m-ATB formulations AF-4 and AF-8 gave wet Tg's that were lower than the dry Tg's by 4° and 22°C, respectively, while the m-ATS formulations AF-11 and AF-20 gave wet Tg's that were 17° and 2°C lower, respectively, than their dry Tg's. Differenes in the rate of drying may account for the wet Tg differences between AF-4 and AF-8 and the differences between AF-11 and AF-20. Even the 22°C difference between wet and dry Tg for AF-4 can be considered excellent when compared to BMI's and epoxies. Some BMI's have wet Tg's nearly 100°C lower than the dry Tg. AF-8 has slightly improved hot/wet properties over AF-4 with a 54.6% retention of room temperature dry modulus at 350°F compared to 48.9% for AF-4. A comparison of the m-ATS formulations shows AF-20 maintains 56.6% of room temperature dry modulus at 450°F compared to only 43.6% for AF-11. Both m-ATS formulations have significantly better hot/wet properties than the m-ATB formulations and are far superior to toughened BMI's.

4.1.2.4 Thermogravimetric Analysis (TGA)

Listed respectively in Tables 3 and 6 are the TGA's for the cured m-ATB and m-ATS formulations run in nitrogen (thermal) and air (thermal oxidative). This data indicates that there is not much variation in thermal or thermal oxidative stability between the formulations and their respective unmodified controls. All of the resins have very favorable polymer decomposition temperatures and exceed the predicted use temperature for the resins by a considerble margin of safety.

4.1.2.5 Compact Tension Fracture Toughness, G_{1c}

The G_{1c}'s are recorded in Table 3 for the m-ATB formulations and Table 6 for the m-ATS formulations. Both the unmodified m-ATB and m-ATS resins have a fracture toughness of 0.2 in-lbs/in^2. AF-4 is tougher than AF-8, with corresponding lower elevated temperature properties than AF-8, while AF-20 is tougher than AF-11 without sacrificing elevated temperature properties as judged by DMTA results. All four formulations show a significant improvement in fracture toughness over the unmodified resins.

4.1.2.6 Tensile Strength, Modulus, and Strain

In Phase 1 of this contract dogbone specimens (Figure 43) were run on AF-4 and AF-8 (Figures 44 and 45) at 325°, 400° and 425°F as a check on DMTA neat resin modulus data. Since the dogbone modulus data compared well with DMTA, only the DMTA was used to obtain Phase 2 modulus data in order to save time and material.

4.2 Task II: Preparation of Prepreg Material

Production quality prepreg with good tack, drape, and handleability was made from all four formulations AF-4, AF-8, AF-11, and AF-20 on Hysol Grafil fiber. Unmodified m-ATB and m-ATS on XAS fiber served as prepreg controls.

4.2.1 Resin Content and Fiber Areal Weight

These properties along with other prepreg physical data are presented in Table 9. The goal has been to produce a neat resin prepreg at a resin content of 33±2% and a fiber areal weight of 145±3 g/m^2.

4.2.2 Volatile Content

AF-4, AF-8, AF-11, and AF-20 are all 100% solids resins containing no solvents. The volatiles present are reactive diluents which cross link during the cure cycle and thus pose no special processing problems for the resins. The volatiles contents obtained are typical of 100% solids prepreg systems containing reactive diluents.

4.2.3 Resins Flow

Typical resin flows for carbon fiber prepreg are in the range of 5-15%. At flows of 6.8 and 8.7 percent for AF-4 and AF-8, respectively, (Table 9) these resins would be considered medium flow systems. The AF-20 formulation of m-ATS is a medium flow system at 8.7% while the AF-11 formulation of m-ATS is a high flow system at 11% (Table 9). Unmodified m-ATS at 11% and unmodified m-ATB at 14.8% are high flow systems and thus susceptible to processing problems such as voids and low resin content in cured laminates. While the high flow of the unformulated resins is due to the low resin viscosity, the high flow of AF-11 is due to its long gel time. Efforts to reduce this gel time under IR&D funding have shown strong promise. AF-4, AF-8, and AF-20 prepreg was easy to process since each resin was a medium flow or "controlled flow" system.

4.2.4 Gel Time

Gel times for the m-ATB, m-ATS, and the four formulations are listed in Table 9. The gel times for AF-4, AF-8, and AF-20 fall within what would be considered an optimum range that is long enough to

provide good fiber wet-out and compaction of prepreg but short enough to prevent excessive resin bleed and thus prevent resin starved laminates. The long gel time of AF-11, however, makes it more difficult to produce high quality laminates due to excessive flow. As mentioned before, efforts to reduce this gel time under IR&D funding have shown strong promise.

4.2.5 Tack

All four formulations AF-4, AF-8, AF-11, and AF-20 pass the tack test described in Section 2.3.2 and would be classified as having good tack. Unmodified m-ATS with an uncured Tg above room temperature produces a boardy prepreg with no tack while on the other extreme unmodified m-ATB is too tacky and, therefore, difficult to use.

4.2.6 Out Time

The out time as measured in Section 2.3.2 is considered the "work life" of the prepreg. This is the time over which the prepreg maintains its tack, drape, and handleability. The unmodified m-ATS has no out time since it failed the tack test while the unmodified m-ATB has an out time exceeding two months. In comparison, epoxies generally have out times in the range of 2-4 weeks and BMI's about 2 weeks maximum. All four formulations AF-4, AF-8, AF-11, and AF-20 have out times of approximately 2 weeks which meets the generally accepted out time requirements of 10-14 days minimum.

4.3 Task III Results: Fabrication, Characterization and Evaluation of Composite Test Panels

4.3.1 Lay-up, Bagging Procedure and Cure/Post Cure Cycle Development

The laminate cure cycles are similar to those established for the preparation of neat resin castings in Task I and are as follows:

For AF-4 and AF-8:

Pressurize to 100 psi.
Heat-up to 235°F at 2-3°F/minute.
Hold at 235°F for 90 minutes.
Heat-up to 355°F at 2-3°F/minute.
Hold at 355°F for 300 minutes.
Cool down at 1-2°F/minute.

For AF-11 and AF-20:

Pressurize to 100 psi.
Heat-up to 250°F at 2-3°F/minute.
Hold at 250°F for 30 minutes.
Heat-up to 355°F at 2-3°F/minute.
Hold at 355°F for 300 minutes.
Cool down at 1-2°F/minute.

The above cure cycles produced high quality void-free laminates as judged by SEM photomicrographic inspection of a polished edge of a cured laminate. Neat resin DMTA experiments on the m-ATB formulations AF-4 and AF-8 and the m-ATS formulations AF-11 and AF-20 demonstrated that post cures of 550°F and 572°F, respectively, gave the best high temperature properties. Slightly lower post cure temperatures, however, produced the highest quality laminates at 511°F and 550°F, respectively, for the m-ATB and m-ATS formulations. These lower post cure temperatures were utilized for the composite testing and data accumulation. All post cures were 240 minutes long. Figure 6 illustrates the "epoxy like" lay-up and bagging autoclave procedure for the prepreg of all four formulations.

4.3.2 Prepreg Fiber Characterization

All composite data was generated from prepreg fabricated from the four formulations and their respective control resins m-ATB and m-ATS applied to Hysol Grafil XAS high strain carbon fiber. The carbon fiber had an average tensile strength of 650 ksi and 616 ksi for the m-ATB and m-ATS based formulations, respectively, and a modulus of 33.5 msi for all applications. The fibers received standard oxidative surface treatments followed by the application of a 1.3% by weight epoxy size ("A" size).

4.3.2.1 Fiber Volume, Density and Void Content

The resin content and/or fiber volume was determined on all laminates fabricated. Due to difficulty in digesting the formulated laminates in nitric acid, the resin content and fiber volume were calculated from the measured densities of the laminate, the cured neat resin, and the fibers. The m-ATB formulations had fiber volumes of $61\pm1\%$ and the m-ATS formulations had fiber volumes of $62\pm2\%$.

Each fabricated laminate was cross sectioned, polished, and examined under the SEM to reveal high quality laminates with void contents under 1%. As a result of the data generated in this section, it may be concluded that the composite mechanical test results in Tables 10 and 11 came from laminates of approximately equivalent quality.

4.3.3 Composite Mechanical Properties Tests

4.3.3.1 Interlaminar Shear Strength

The interlaminar shear depends entirely on the shear strength between the fiber and matrix. This property will fall off dramatically at higher temperatures (above matrix Tg) and is therefore a good screening test to assess the high temperature properties of the formulations. The interlaminar shear results of the m-ATB formulations AF-4 and AF-8 show a substantial improvement over the unmodified m-ATB at room temperature while the m-ATS formulations

AF-11 and AF-20 show a smaller improvement over the unmodified m-ATS (Tables 10 and 11). AF-4 and AF-8 give improvements of 42.7% and 29.1%, respectively, over m-ATB while AF-20 shows only a 16.0% increase over m-ATS and AF-11 is virtually no greater in interlaminar shear strength than m-ATS at room temperature. In absolute terms, however, AF-20 has a higher shear strength than AF-8 as expected from the higher shear strength of m-ATS over m-ATB (the even higher shear strength of AF-4 is less significant in view of its lower values at elevated temperatures). The m-ATB formulations AF-4 and AF-8 elevated 0 degree interlaminar shear strengths parallel the DMTA modulus results and as expected AF-8 has better shear strengths than AF-4 at 350°F and 450°F. AF-20 also follows the DMTA modulus results, but the interlaminar shear strength of AF-11 declines faster than would be expected based on neat resin data. This can be attributed to the long gel time and high resin flow resulting in prepreg processing difficulties and subsequent lowering of composite quality. Similar problems were encountered in the early stages of AF-20 development after the completion of AF-11 work but a solution was found. It would be possible to apply a similar soluton to AF-11 with the probable result being an increase in high temperature properties. The m-ATB formulations AF-4 and Af-8 retain 24.8% and 39.4% of their respective ambient temperature shear strengths at 450°F while the m-ATS formulation AF-20 retains 38.6%. Unmodified m-ATB retains 35.5% of ambient shear strength while m-ATS retains 46.4% at 450°F.

Despite its low moisture absorption the unmodified m-ATB has very poor retention of dry ambient shear strength for the wet elevated temperature conditions. AF-8, however, has an excellent 45.8% retention at 350°F and 33.8% retention at 425°F. AF-4 is lower with 30.6% and 19.7% retention at 350°F and 425°F, respectively. The m-ATS formulations tend to do even better than the m-ATB formulations at 350°F wet conditions with 52.4% and 47.6% retentions of dry ambient shear for AF-11 and AF-20,respectively. At 425°F wet conditions AF-11 and AF-20 maintain 31.7% and 24.1% of ambient dry values which fall between the AF-8 and AF-4 retentions.

4.3.3.2 0 Degree Compression Strength and Modulus

Of particular interest are the changes in mechanical behavior due to moisture exposure which have been observed in such matrix dominated properties as compression strength and shear modulus. These changes in mechanical behavior are more pronounced at elevated temperatures. Because significant differences in compression properties are caused by changes in temperature and by moisture absorption

in the composite laminate, the 350°F/wet and 425°F/wet compression strengths were determined in the 0 degree direction to show the ability of the matrix to stabilize the graphite filaments. The results are shown in Tables 10 and 11.

As expected from DMTA results, AF-8 has better 350°F/wet and 425°F/wet retention of dry ambient compressive strength than AF-4 with values at 350°F of 63.3% versus 47.5% and at 425°F of 58.9% versus 44.8%, respectively. AF-20 has both the best retention (71.2%) and absolute value (131.0 ksi) of the four formulations at 350°F/wet which would be expected based on DMTA and the overall hot/wet compressive performance of unmodified m-ATS versus m-ATB. The 425°F/wet value is lower than expected for AF-20 based on DMTA and m-ATS values while the overall performance of AF-4 and AF-8 is lower than anticipated from their low equilibrium moisture absorptions. Even so, the hot/wet compressive values compare favorably with toughened BMI's.

Both of the formulated m-ATB resins have dry compression strengths greater than the m-ATB control at all temperatures. AF-20 dry elevated temperature compression strengths are comparable to m-ATS in absolute terms and superior in retention of ambient values.

4.3.3.3 0 Degree Tension Strength and Modulus

In the composite the tensile strength and modulus are largely dominated by the fiber properties so that the composite tensile strength provides a measure of the translation of these properties into the composite. Present ultimate tensile strengths are limited by the severity and frequency of fiber flaws and the matrix must be able to redistribute the load under conditions of high shear caused by isolated fiber breaks. The inability of the matrix to redistribute the load is magnified at lower temperatures. Based on the fiber volume of the test samples AF-4, AF-8, and AF-20 have excellent translations of the fiber strength into the composite with 74%, 88%, and 80% translations at -67°F. In addition, all three of these formulations exceed the composite tensile strengths of their respective unmodified controls, ATB and ATS, at both room temperature and -67°F. The data is tabulated in Tables 10 and 11.

4.3.3.4 0 Degree Flexure Strength and Modulus

When a specimen is tested in flexure one face is under compression while the other is in tension so that the flexure strength and modulus are a good measure of overall composite properties. The elevated temperature dry and wet

flexure properties should parallel the neat resin DMTA results. Both AF-11 and AF-20 have a slightly higher flexure modulus than m-ATS as expected while AF-4 and AF-8 have a slightly lower flexure modulus than m-ATB. The AF-4 and AF-8 results could be due in part to variations in fiber volume. However, the room temperature flexure strengths of AF-4 and AF-8 are 58% and 61% higher than m-ATB while AF-11 and AF-20 are 5% and 14% higher than m-ATS. At 450°F, AF-4 and AF-8 maintain 46% and 62% of ambient temperature flexure strength while AF-20 maintains 47%. The m-ATB and m-ATS retains 66% and 65% but in absolute terms both AF-4 and AF-8 exceed m-ATB at 450°F. Test results are recorded in Tables 10 and 11.

As expected from its low moisture absorption, m-ATB has the best retention of dry ambient flexure strength at wet elevated temperature conditions with 71% and 56% retention at 350°F/wet and 425°F/wet. At 350°F/wet both AF-4 and AF-8 retain 57% of dry ambient flexure strength while at 425°F/wet AF-8 is superior to AF-4 with 44% and 32% retention, respectively. AF-20 has only slightly lower retention than m-ATS at 48% versus 53% for 350°F/wet and 43% versus 49% for 425°F/wet. In absolute terms AF-20 has slightly better wet elevated flexure strength than m-ATS at both 350°F/wet and 425°F/wet while AF-4 and AF-8 are superior to m-ATB at 350°F/wet with m-ATB falling between AF-4 and AF-8 at 425°F/wet.

4.3.3.5 Edge Delamination Strength

This test is used to measure the interlaminar fracture toughness. The lay up utilized, $[(+30,-30)_2,90]_S$ gives a fracture toughness, Gc, which is 57% Glc or crack opening and 43% G2c or shear. The formulated m-ATS resins AF-11 and AF-20 give values 81% and 25% greater than the unmodified m-ATS. AF-4 and AF-8 show dramatic improvements over m-ATB with increases of 119% and 125%. The results appear in Tables 10 and 11.

4.3.3.6 90 Degree Flexure

In 90 degree flexure, a matrix dominated property, one face is under tension while the other is under compression. The failure may occur in the matrix or at the fiber/matrix interface. Assuming that the failure occurs in the matrix, this test yields data on the relative toughness of a resin. The unmodified m-ATB has the lowest strength and strain (measured on the tensile face) which would be expected based on the brittle nature of the resin and the poor fiber/resin interface shown by SEM. Both AF-4 and AF-8 show a 300% improvement over m-ATB in strain and improvements of 161% and 203%, respectively, in strength.

The unmodified m-ATS has strength and strain 125% and 168% greater than m-ATB. Consequently, AF-20 shows a less dramatic 33% improvement in strength and 19% improvement in strain over m-ATS. In absolute terms AF-8 and AF-20 are approximately the same in strength while AF-4 is slightly less. These results appear in Tables 10 and 11.

4.3.3.7 Uniform Double Cantilever Beam Test

From the uniform double cantilever beam test the mode I critical strain energy release rate, G1c, can be calculated. The G1c gives a measure of the fracture toughness of the various formulated resins. As expected m-ATB gives the lowest value at 1.1 in-lbs/in^2. AF-4 and AF-8 gave increases in toughness of 91% and 64%, respectively. AF-11 and AF-20 are 43% and 32% tougher than m-ATS but in absolute terms show greater toughness than either AF-4 or AF-8. The data is recorded in Tables 10 and 11.

4.3.3.8 ± 45 Degree In-Plane Wet Shear Strength and Modulus

This test was run for two reasons. First, this test measures in-plane shear and will point out weaknesses in the fiber/resin interface. The wet conditions magnify poor interfacial bonding. Second, the shear modulus, G12, is a matrix dominated property and therefore a good measure of the hot/wet performance of the matrix resin when it is carried out under those conditions. AF-20 has the highest in-plane shear modulus at 350°F/wet which is not surprising given that m-ATS is 38% greater than m-ATB at 350°F/wet. Although m-ATB maintains 86% of its 350°F/wet modulus at 425°F/wet and m-ATS maintains only 65%, AF-20 has a better retention than either AF-4 or AF-8 with retentions of 59%, 50%, and 40%, respectively. In absolute terms AF-20 far exceeded both AF-4 and AF-8 at 425°F/wet with the two m-ATB formulations only 65% and 54% the value of AF-20. The AF-20 425°F/wet shear strength is also higher than the AF-4 and AF-8 values in absolute terms. In relative terms AF-20 retains 80% of its 350°F/wet shear strength at 425°F/wet while AF-4, AF-8, and AF-20 all have greater in-plane shear strength at both 350°F/wet and 425°F/wet conditions than their respective unmodified controls m-ATB and m-ATS. Test results are shown in Tables 10 and 11.

4.3.3.9 Thermal Aging and Thermal Spikes

Both the unmodified m-ATB and m-ATS composite specimens showed severe microcracking under microscopic examination after 25 hours of thermal aging at 350°F plus 264 hours at 275°F. The m-ATS formulations AF-11 and AF-20 showed some minor microcracking while the m-ATB formulations AF-4 and AF-8 had no microcracking. All of the specimens performed

well in the thermal spike test with m-ATB, m-ATS, AF-11 and AF-20 all showing only 0.06% weight gain over the control laminates while AF-4 and AF-8 showed 0.08% and 0.12% respectively.

Table 1 Neat Resin Test Matrix

Test	Room Temperature Dry	Room Temperature Wet
Uncured Tg	3	
DSC-uncured resin	3	
Rheometric Cure Profile	3	
DMTA (Tg, Flex modulus)	3	
Moisture Absorption:[a]		
Weight Change		3
DMTA (WET Tg)		3
TGA:		
Air	3	
Nitrogen	3	
Tensile Dogbones	5	
Compact Tension, G_{1c}	5	

a. Immersed in 160°F water until weight gain over 7 day period was less than 0.04% of the original.

Table 2 Detailed Laminate Test Matrix

Test	Test Temperature Dry				Test Temperature Water Saturated[a]			Total Specimens
	-67°F	Room	350°F	450°F	Room	350°F	425°F	
0-deg Tension	5	5	-	-	-	-	-	10
0-deg Flexure	-	5	5	5	5	5	5	30
0-deg Interlaminar Shear	-	5	5	5	5	5	5	30
90-deg Flexure	-	5	-	-	-	-	-	5
Edge Delamination (±30°, 90°)	-	5	-	-	-	-	-	5
In-plane Shear	-	-	-	-	-	5	5	10
0-deg Compression	-	5	5	5	-	5	5	25
Double Cantilever Beam	-	5	-	-	-	-	-	5
Tg	-	1	-	-	1	-	-	2
Thermal Spikes	-	-	-	-	-	3	-	3
Thermal Aging	-	-	3	-	-	-	-	3
Void Content	-	5	-	-	-	-	-	5
Fiber Volume	-	5	-	-	-	-	-	5
Density	-	5	-	-	-	-	-	5

a. Immersed in 160°F water until weight gain over a 7 day period was less than 0.04% of the original.

Table 3 Neat Resin Properties: IR&D m-ATB Formulations

NEAT RESIN PROPERTY	FORMULATION									
	m-ATB	AF 1	AF 2	AF 3	AF 4	AF 5	AF 6	AF 7	AF 8	AF 9
DSC, °c										
Onset	184.7	190	200	210	208.1	202	209	211	207.9	212
Peak Exotherm	237	236	237	249	254.9	102	100	253	254.7	253
Uncured Tg, °C	14°	--	--	--	3°	--	--	--	-6°	--
Minimum Viscosity, (Poise) °C	0.20 at 156°	257 at 154°	11.4 at 150°	15 at 180°	8.4 at 179°	21 at 152°	31 at 173°	180 at 187°	14.6 at 171°	6.8 at 182°
Tg, Dry °C	260	232	220	192, 257	260	230, 170	230	196, 252	273	198, 257
Young's Modulus[a], 23°C Ksi	445	435	470	508	489	417	475	520	467	552
Eq. Moisture Absorption, %	.60	3.9	1.46	1.63	1.40	7.9	1.25	1.54	1.43	1.84
Tg, Wet °C [b]	252	160	215	240	258	168	215	225	251	234
Young's Modulus, [a],[b]23°C,Ksi	376	300	233	302	406	199	355	406	434	245
TGA, °C at 10% wt. loss										
Air	365	355	357	357	397	331	344	366	350	368
N_2	446	355	384	391	458	383	371	384	421	384
G_{1c}, in lbs/in^2	0.2	0.63	1.25	1.03	0.94	0.52	0.95	1.30	0.65	0.33

a. Calculated using Young's Modulus (RT) from DMTA
b. Specimens immersed in 160°F water bath till equilibrium (about 14 days)

Table 4 DMTA Modulus/Temperature Profile
IR&D m-ATB Formulations

MODULUS (KSI)

FORMULATION	RT	350°F	400°F	425°F	450°F
m-ATB	445	342	289	246	191
AF-1	435	61.9	28.3	21.5	15.2
AF-2	470	132	38.2	25.8	17.1
AF-3	508	310	183	183	98.1
AF-4	489	333	243	187	126
AF-4/525°F PC	470	283	205	160	120
AF-4/550°F PC	508	393	320	263	205
AF-5	417	22.5	11.0	6.19	5.65
AF-6	475	77.9	29.0	18.3	12.4
AF-7	520	340	183	126	89.5
AF-7/525°F PC	494	288	166	117	80.1
AF-7/550°F PC	469	264	152	110	69.5
AF-8	467	329	263	209	150
AF-8/525°F	443	290	225	183	135
AF-8/550°F	428	296	252	215	171
AF-9	552	400	179	137	103

Table 5 DMTA Wet Modulus/Temperature Profile
IR&D m-ATB Formulations

FORMULATION	RT	300°F	350°F	400°F	425°F
m-ATB	376	297	261	209	162
AF-1	300	144	87	74	-
AF-2	233	170	76	28	16
AF-3	302	215	162	114	88
AF-4	406	302	239	165	124
AF-5	199	57	25	7.6	-
AF-6	355	261	179	90	60
AF-7	406	161	104	64	49
AF-8	434	312	255	177	120
AF-9	245	154	108	72	57

Table 6 Neat Resin Properties: IR&D m-ATS Formulations

NEAT RESIN PROPERTY	FORMULATION											
	m-ATS	AF 10	AF 11	AF 12	AF 13	AF 14	AF 15	AF 16	AF 17	AF 18	AF 19	AF 20
DSC, °C												
Onset	204.3	213.5	213.6	214.7	214.7	98.3	179.6	218.7	213.4	206.0	228.4	198.9
Peak Exotherm	227.5	252.3	240.7	252.5	258.0	243.7	237.5	250.2	255.7	241.7	253.3	218.9
Uncured Tg, °C	25°	--	5°	--	--	--	--	--	--	--	--	11°
Minimum Viscosity, (Poise) °C	.7 at 120°	4.3 at 184°	7.9 at 186°	14.6 at 178°	87.3 at 162°	22.5 at 176°	5.8 at 169°	15.2 at 164°	2.66 at 181°	2.2 at 161°	4.4 at 180°	19.8 at 171°
Tg, Dry °C	331	300	292	330	301	205, 306	205 319	298	205, 310	197, 283	310	302
Young's Modulus, [a]23°C, Ksi	454	466	456	572	418	459	426	346	473	467	446	544
Eq. Moisture Absorption, %	1.85	1.80	2.00	2.35	2.07	1.81	1.66	1.43	2.55	1.45	1.51	2.04
Tg, Wet °C [b]	321	299	288	320	305	309	311	282	296	310	297	290
Young's Modulus, [a,b]23°C,Ksi	430	591	529	389	453	366	447	426	364	469	432	524
TGA, °C at 10% wt. loss												
Air	438	414	407	419	390	378	376	390	400	419	427	399
N_2	456	462	481	470	427	420	421	457	451	460	470	469
G_{1c}, in lbs/in^2	0.22	0.27	0.65	0.27	0.59	0.41	0.60	0.24	0.32	0.44	0.23	0.80

a. Calculated using Young's Modulus (RT) from DMTA
b. Specimens immersed in 160°F water bath till equilibrium (about 14 days)

Table 7 DMTA Modulus/Temperature Profile: IR&D m-ATS Formulations

FORMULATION	MODULUS (KSI)						
	RT	300°F	350°F	400°F	425°F	450°F	500°F
m-ATS/265°C P.C.	403	336	319	304	294	283	256
m-ATS/300°C P.C.	515	438	409	373	350	324	258
m-ATS	454	372	352	333	324	308	274
AF-10	466	376	359	338	328	315	281
AF-11	456	377	361	336	322	302	223
AF-12	572	458	406	331	293	258	190
AF-13	418	345	325	292	263	234	169
AF-14	459	368	342	284	257	236	194
AF-15	426	337	302	209	174	141	101
AF-16	519	429	408	385	369	349	278
AF-17	473	390	376	326	297	276	232
AF-18	467	395	382	324	299	270	171
AF-19	466	390	373	354	341	325	282
AF-20[a]	544	459	439	416	399	380	313

Postcure 288°C except where otherwise noted.

a. Postcure 300°C

Table 8 Wet DMTA Modulus/Temperature Profile: IR&D m-ATS Formulations

FORMULATION	MODULUS (KSI)						
	RT	300°F	350°F	400°F	450°F	475°F	500°F
m-ATS/300°C P.C.	487	376	356	335	303	283	259
m-ATS	430	335	321	306	283	269	251
AF-10	591	470	443	420	386	368	337
AF-11	529	392	360	318	241	198	154
AF-12	389	317	294	257	211	190	159
AF-13	453	352	333	310	273	246	207
AF-14	366	263	224	185	149	135	115
AF-15	447	343	296	217	147	124	100
AF-16	426	340	318	295	255	217	174
AF-17	361	236	231	222	186	162	136
AF-18	469	368	333	270	185	140	93
AF-19	432	333	317	295	259	232	194
AF-20[a]	524	416	391	361	308	259	209

Postcure 288°C except where otherwise noted.

a. Postcure 300°C

Table 9 Prepreg Physical Data

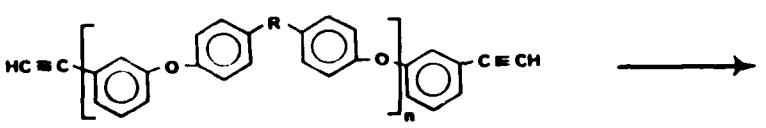
PREPREG PHYSICAL PROPERTIES	FORMULATION					
	m-ATB	m-ATS	AF-4	AF-8	AF-11	AF-20
Fiber Areal Weight, g/m^2	145 ± 4	145 ± 4	145 ± 4	145 ± 4	145 ± 4	145 ± 4
Resin Content, percent weight	30	32 ± 2	31.9	34.9	36 ± 3	30 ± 2
Volatile Content, percent weight	0.68	1.7	0.79	0.65	6.8	3.9
Flow, percent weight	14.8	11	6.8	8.7	11	8.7
Gel Time at 170°C, minute	19	18	7	7	50	16
Out-time at 75°F	>2 months	None	3 weeks	3 weeks	2 weeks	2 weeks
Tack (shelf adhesion)	Pass/Very Good	None	Pass/Good	Pass/Good	Pass/Good	Pass/Good

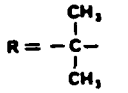
Table 10 Composite Mechanical Properties: m-ATB

TEST	M-ATB		AF-4		AF-8	
	Strength, MPa(Ksi)	Modulus, GPA(Msi)	Strength, MPa(Ksi)	Modulus, GPa(Msi)	Strength, MPa(Ksi)	Modulus, GPa (Msi)
0-deg. Tension						
-67°F	1613 (234)	144 (20.9)	2020 (293)	134 (19.5)	2289 (332)	143 (20.8)
R.T.	1696 (246)	142 (20.6)	1993 (289)	134 (19.5)	2220 (322)	143 (20.8)
0-deg. Flexure						
R.T.	1103 (160)	126 (18.3)	1744 (253)	120 (17.4)	1779 (258)	121 (17.5)
R.T./Wet	752 (109)	125 (18.1)	1606 (233)	127 (18.4)	1689 (245)	121 (17.6)
350°F	855 (124)	123 (17.8)	1282 (186)	114 (16.6)	1510 (219)	122 (17.7)
350°F/Wet	779 (113)	128 (18.6)	993 (144)	100 (14.5)	1007 (146)	117 (16.9)
425°F/Wet	614 (89)	115 (16.7)	558 (81)	91 (13.2)	786 (114)	108 (15.6)
450°F	731 (106)	112 (16.3)	800 (116)	101 (14.7)	1110 (161)	113 (16.4)
0-deg. Interlaminar Shear						
R.T.	75.8 (11.0)		108 (15.7)		97.9 (14.2)	
R.T./Wet	31.7 (4.6)		103 (15.0)		106 (15.4)	
350°F	33.1 (4.8)		47.6 (6.9)		55.2 (8.0)	
350°F/Wet	18.6 (2.7)		33.1 (4.8)		44.8 (6.5)	
425°F/Wet	13.8 (2.0)		21.4 (3.1)		33.1 (4.8)	
450°F	26.9 (3.9)		26.9 (3.9)		38.6 (5.6)	
Edge Delamination Strength, $[(+30,-30)_2,90]_S$	71.0 (10.3)		156 (22.6)		160 (23.2)	
90-deg. Flexure	26.4 (3.83)	strain .25%	68.9 (10.0)	strain 1.0%	80.0 (11.6)	strain 1.0%
In-plane Shear, $\pm 45°$ Tension						
350°F/Wet	60.1 (8.71)	2.00 (.29)	108 (15.7)	2.34 (.34)	117 (17.0)	2.41 (.35)
425°F/Wet	48.3 (7.01)	1.72 (.25)	57.2 (8.3)	1.17 (.17)	80.0 (11.6)	.97 (.14)
0-deg. Compression						
R.T.	855 (124)		1538 (223)		1241 (180)	152 (22.1)
350°F	758 (110)		986 (143)		855 (124)	
350°F/Wet	669 (97)		731 (106)		786 (114)	
425°F/Wet	345 (50)		689 (100)		731 (106)	
450°F	572 (83)		841 (122)		724 (105)	
Double Cantilever Beam G_{IC}, J/m^2 (in-lbs/in^2)	193 (1.1)		368 (2.1)		315 (1.8)	
Thermal Aging	severe microcracking		no microcracking		no microcracking	
Thermal Spikes, % wt.gain	0.06		0.08		0.12	

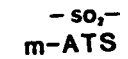
Table 11 Composite Mechanical Properties: m-ATS

TEST	M-ATS Strength, MPa (Ksi)	M-ATS Modulus, GPa (Msi)	AF-11 Strength, MPa (Ksi)	AF-11 Modulus, GPa (Msi)	AF-20 CATALYST Strength, MPa (Ksi)	AF-20 CATALYST Modulus, GPa (Msi)
0-deg. Tension						
-67°F	2016 (292.4)	141 (20.4)	1737 (252.0)	132 (19.1)	2111 (306.1)	141 (20.4)
R.T.	2126 (308.4)	137 (19.8)	1855 (269.0)	138 (20.4)	2256 (327.2)	139 (20.2)
0-deg. Flexure						
R.T.	1628 (236.1)	121 (17.5)	1711 (248.1)	122 (17.7)	1849 (268.1)	121 (17.6)
R.T./Wet	1478 (214.3)	117 (16.9)	1595 (231.3)	117 (16.9)	1629 (236.2)	121 (17.6)
350°F	1273 (184.6)	125 (18.2)	1356 (196.7)	123 (17.8)	1439 (208.7)	121 (17.5)
350°F/Wet	856 (124.2)	114 (16.5)	652 (94.6)	104 (15.1)	889 (129.0)	111 (16.1)
425°F/Wet	799 (115.9)	103 (14.9)	233 (33.8)	35.2 (5.1)	801 (116.2)	80.7 (11.7)
450°F	1064 (154.3)	126 (18.3)	487 (70.6)	72.4 (10.5))	866 (125.6)	117 (16.9)
0-deg. Interlaminar Shear						
R.T.	86.2 (12.5)		86.9 (12.6)		100 (14.5)	
R.T./Wet	66.1 (9.6)		60.7 (8.8)		80.7 (11.7)	
350°F	55.2 (8.0)		56.5 (8.2)		68.3 (9.9)	
350°F/Wet	42.1 (6.1)		45.5 (6.6)		47.6 (6.9)	
425°F/Wet	35.9 (5.2)		27.6 (4.0)		24.1 (3.5)	
450°F	40.0 (5.8)		22.1 (3.2)		38.6 (5.6)	
Edge Delamination Strength, $[(+30,-30)_2,90]_S$	55.8 (8.1)		101 (14.7)		69.6 (10.1)	
90-deg. Flexure	59.3 (8.6)	strain = .67%	53.0 (7.7)	strain = 1.06%	78.6 (11.4)	strain = .8%
In-plane Shear, ±45° Tension						
350°F/Wet	76.5 (11.1)	2.76 (0.40)	93.1 (13.5)	2.62 (0.38)	101 (14.6)	3.03 (0.44)
425°F/Wet	70.3 (10.2)	1.79 (0.26)	68.3 (9.9)	0.97 (0.14)	80.7 (11.7)	1.79 (0.26)
0-deg. Compression						
R.T.	1423 (206.4)	130 (18.9)	1335 (193.6)	134 (19.4)	1269 (184.1)	130 (18.9)
350°F	1047 (151.9)	140 (20.3)	1091 (158.2)	125 (18.1)	1076 (156.1)	134 (19.5)
350°F/Wet	1013 (146.9)	124 (18.0)	854 (123.9)	137 (19.8)	903.2 (131.0)	128 (18.6)
425°F/Wet	821 (119.1)	126 (18.3)	206 (29.8)		622.6 (90.3)	120 (17.4)
450°F	928 (134.6)	137 (19.8)	243 (35.2)	137 (19.9)	845.3 (122.6)	139 (20.1)
Double Cantilever Beam G_{IC}, J/m^2 (in-lbs/in^2)	490 (2.8)		700 (4.0)		648 (3.7)	
Thermal Aging	severe microcracking		minor microcracking		minor microcracking	
Thermal Spikes, % wt.gain	0.06		0.06		0.06	

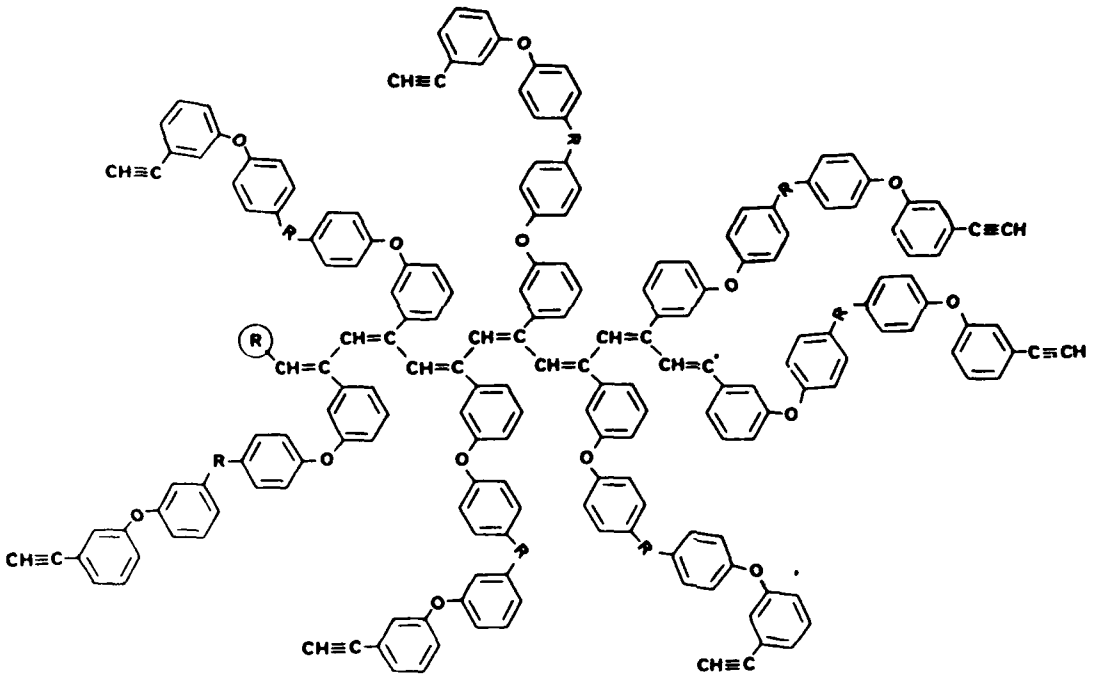




m-ATB



m-ATS



early stages of cure

FIGURE 1 AT RESIN CHEMICAL STRUCTURE AND CURE MECHANISM

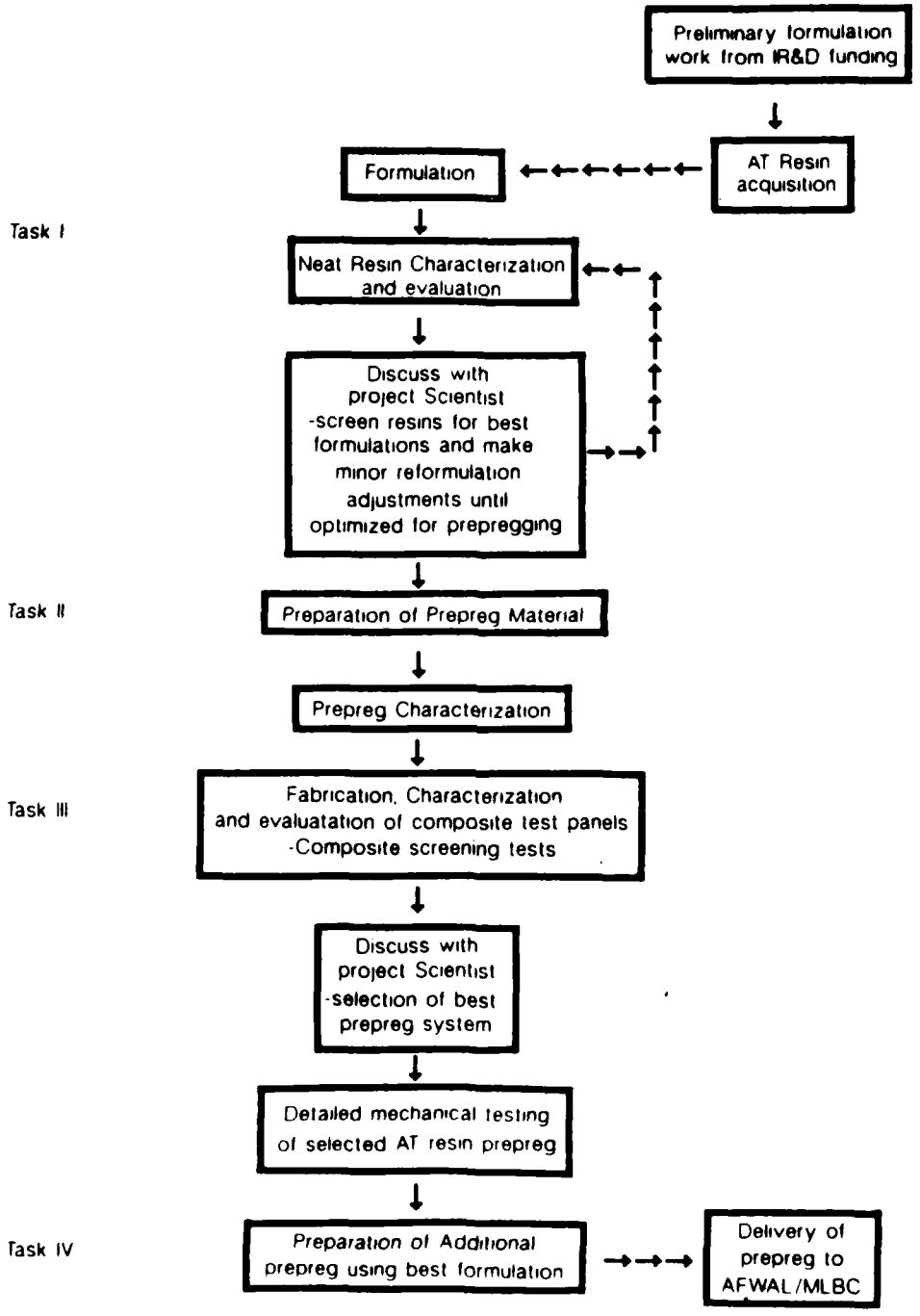
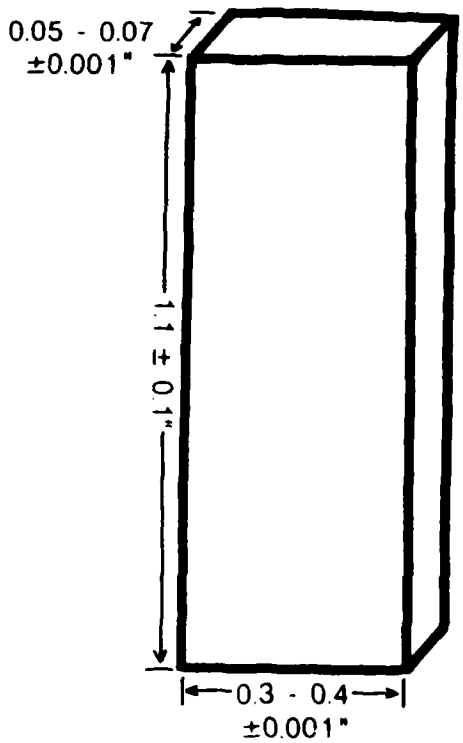


FIGURE 2 PROGRAM FLOW CHART



note: all dimensions in inches. Acutal dimensions should be recorded to the nearest 0.001 inch

Test type: Dual cantilever bending

Mode: Temperature sweep at 3°/min dry, 5° min wet

% strain: 6.25

Frequency: 1 Hz

Temperature Range: RT to matrix Tg + 50°F

Flexural modulus (E') and loss tangent (tan δ) as a function of temperature.

FIGURE 3 NEAT RESIN DMTA SPECIMEN

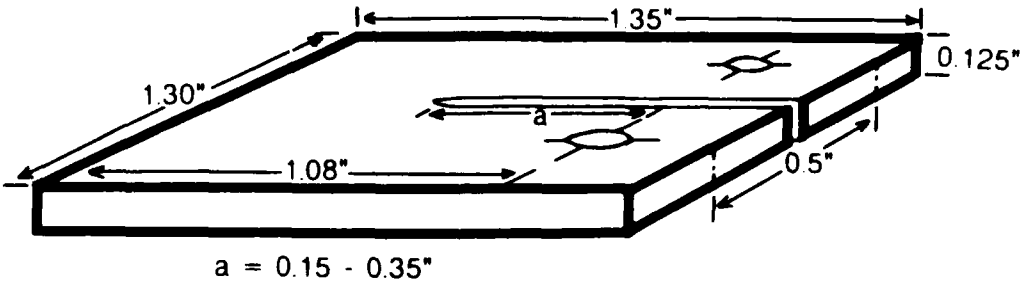


FIGURE 4 FRACTURE TOUGHNESS, G1c, COMPACT TENSION SPECIMEN

Formulation_____ Fiber Areal Wt._____

Prepreg Physical Properties

1. Resin Content, percent weight _____
2. Volatile Content, percent weight _____
3. Flow, percent weight _____
4. Gel time, minutes _____
5. Out-time at RT _____
6. Tack (self adhesion) _____

FIGURE 5 PREPREG PHYSICAL DATA SHEET

1. 1-inch minimum width with connection to vacuum source - at one corner of the layup, place a single fiberglass yarn between the edge of the layup and the edge breather to allow evacuation of air from the layup. Additional yarns may be required on larger parts to provide adequate removal of trapped air.

2. If fiberglass is used for surface breathers, it shall be net trimmed to the edge of the layup. At one position, connect the surface breather to the edge breather using a single fiberglass yarn - if Airweave SS is used for surface breathers, it may extend to connect with the edge breather.

3. FEP extends to centerline of edge breather.

4. Pressure plate 0.20 inch thick minimum.

5. Surface breather required unless pressure plate is used, then breather is not required.

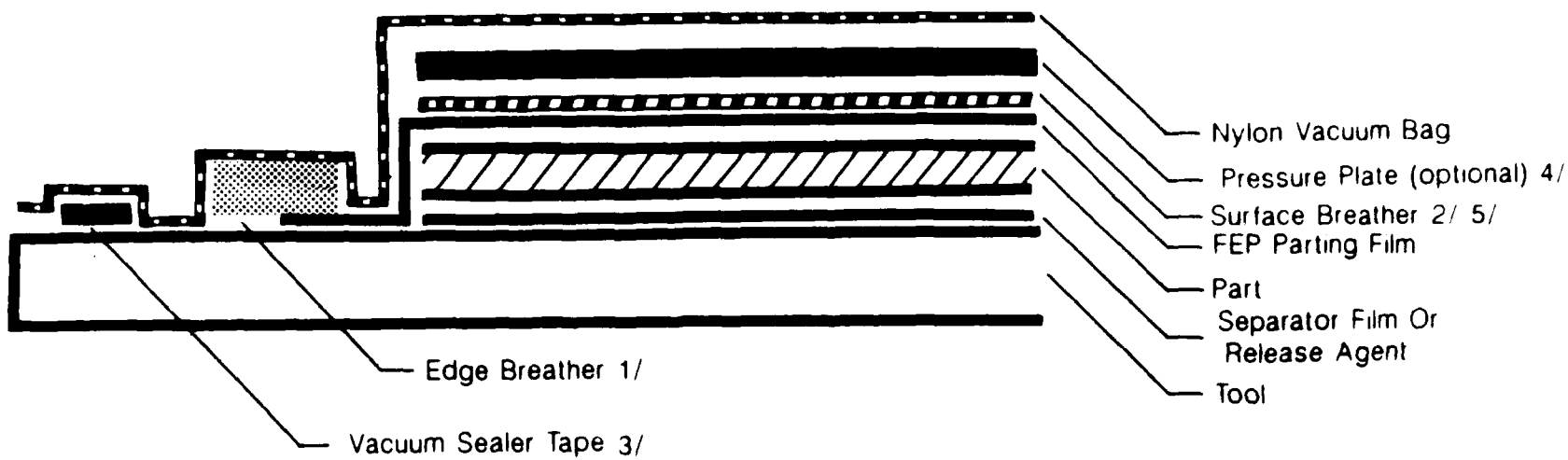


FIGURE 6 LAY-UP, BAGGING PROCEDURE

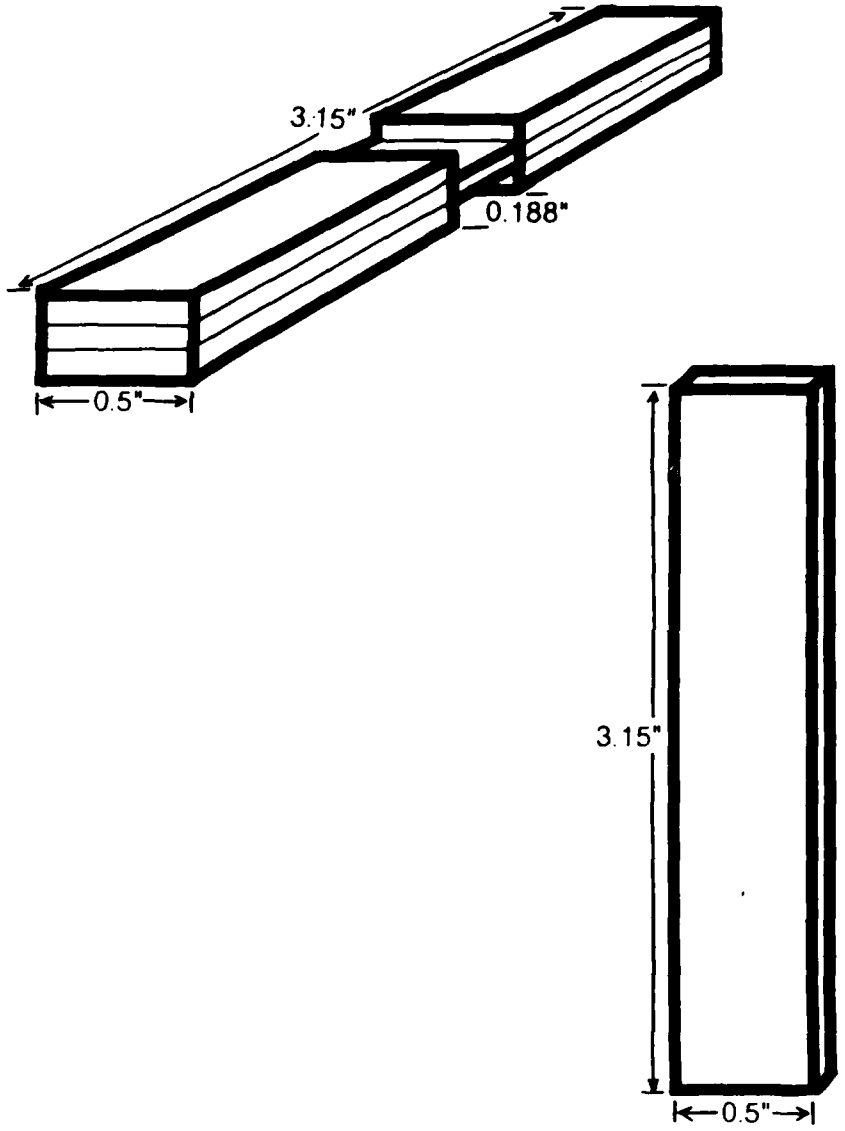


FIGURE 7 0-DEG. COMPRESSION STRENGTH AND WEIGHT CHANGE SPECIMEN

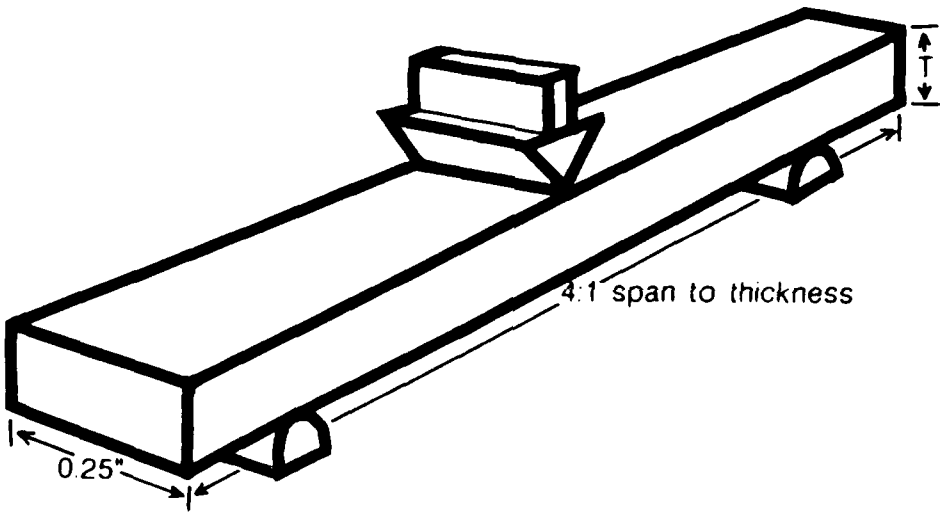


FIGURE 8 0-DEG. INTERLAMINAR SHEAR

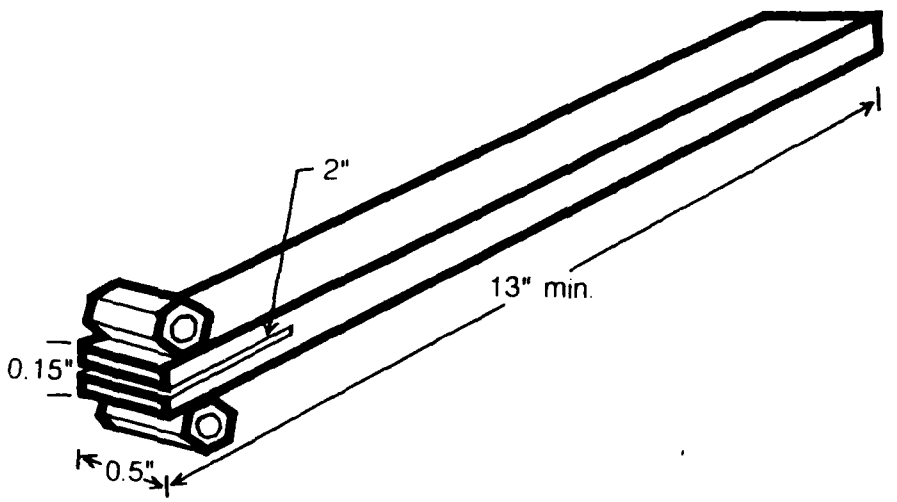
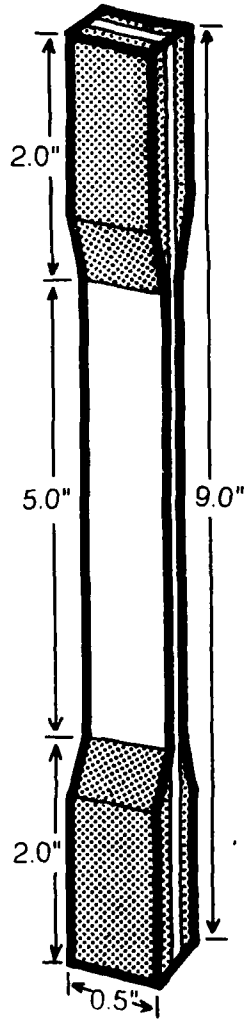


FIGURE 9 UNIFORM DOUBLE CANTILEVER BEAM SPECIMEN FOR G1c



All lengths to ± 0.1"

WIDTH AND THICKNESS MEASUREMENTS TO ± 0.001"

FIGURE 10 TAPE TABBED 0-DEG. TENSION SPECIMEN

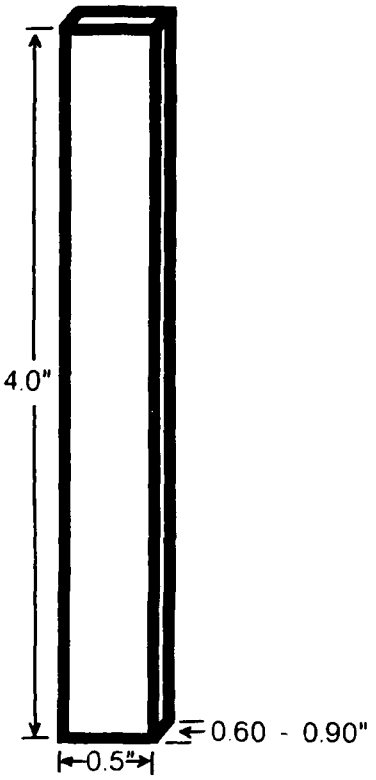
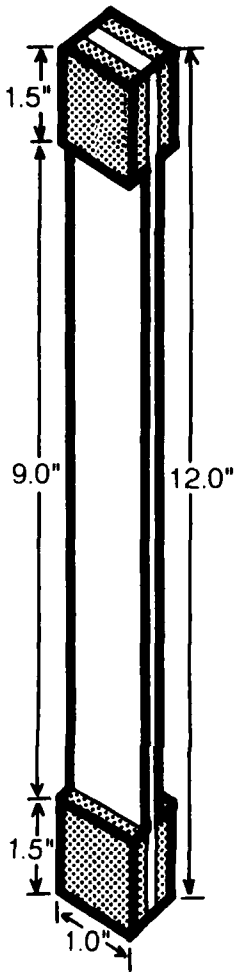


FIGURE 11 0-DEG. AND 90-DEG. FLEXURE SPECIMEN



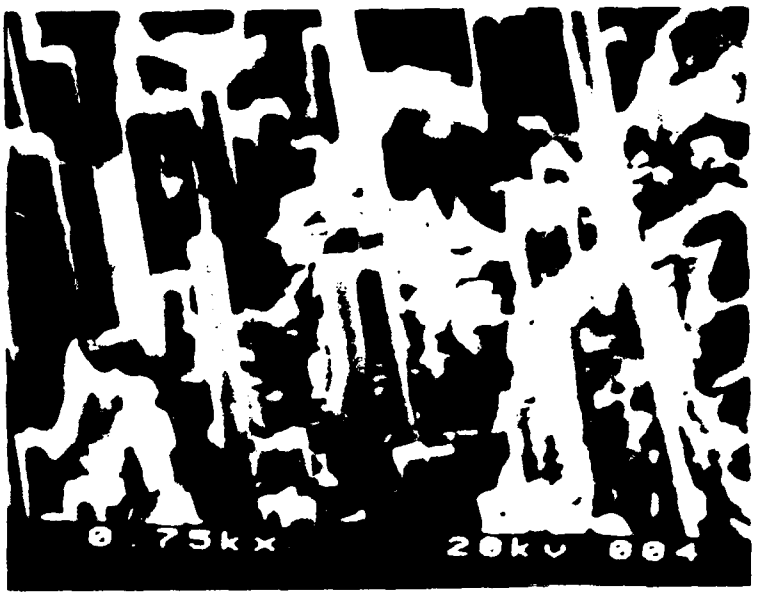
ply lay-up:$[(+30,-30)_2,90]_s$

FIGURE 12 EDGE DELAMINATION TENSION SPECIMEN

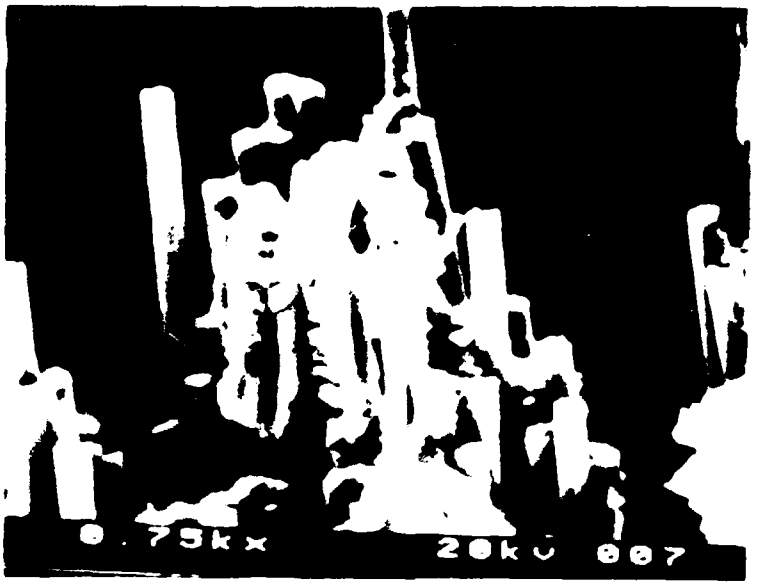
SEM PHOTOMICROGRAPH OF m-ATS ON XAS CARBON FIBER

SEM PHOTOMICROGRAPH OF m-ATB ON XAS CARBON FIBER

FIGURE 13



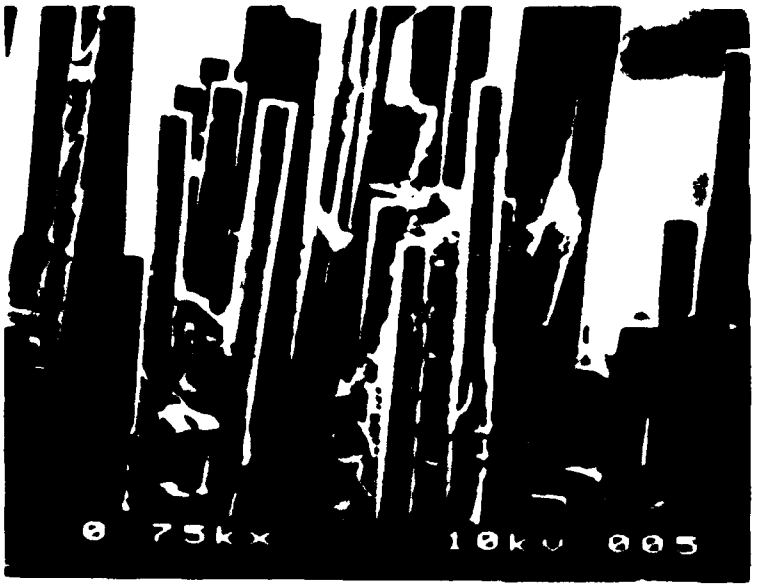
SEM PHOTOMICROGRAPH OF AF-8 ON XAS CARBON FIBER



SEM PHOTOMICROGRAPH OF AF-4 ON XAS CARBON FIBER

FIGURE 14

SEM PHOTOMICROGRAPH OF AF-20 ON XAS CARBON FIBER



SEM PHOTOMICROGRAPH OF AF-11 ON XAS CARBON FIBER

FIGURE 15

SEMI-LOGARITHMIC 4 CYCLES X 100 DIVISIONS RHEOMETRICS, INC. R-2438-5

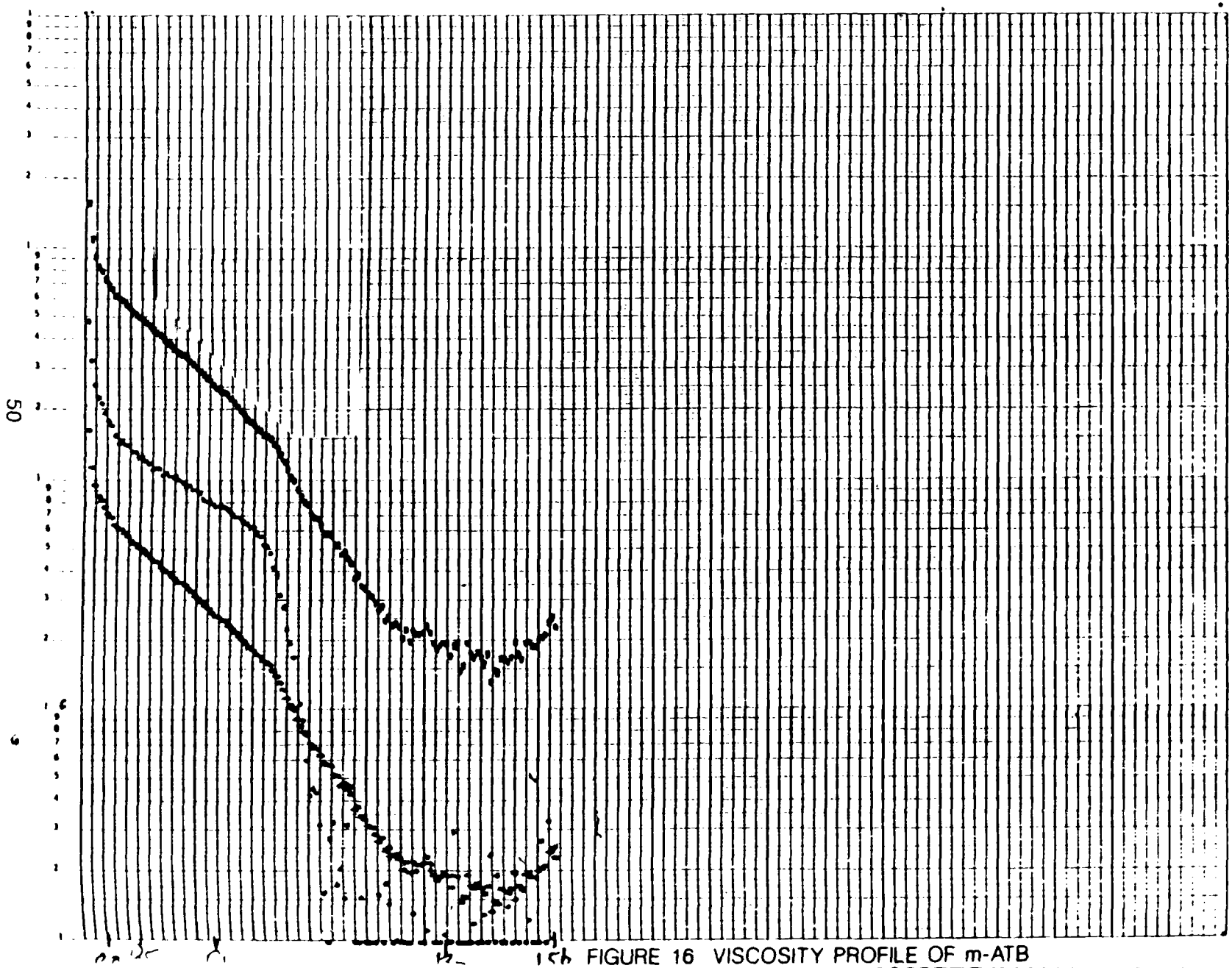


FIGURE 16 VISCOSITY PROFILE OF m-ATB

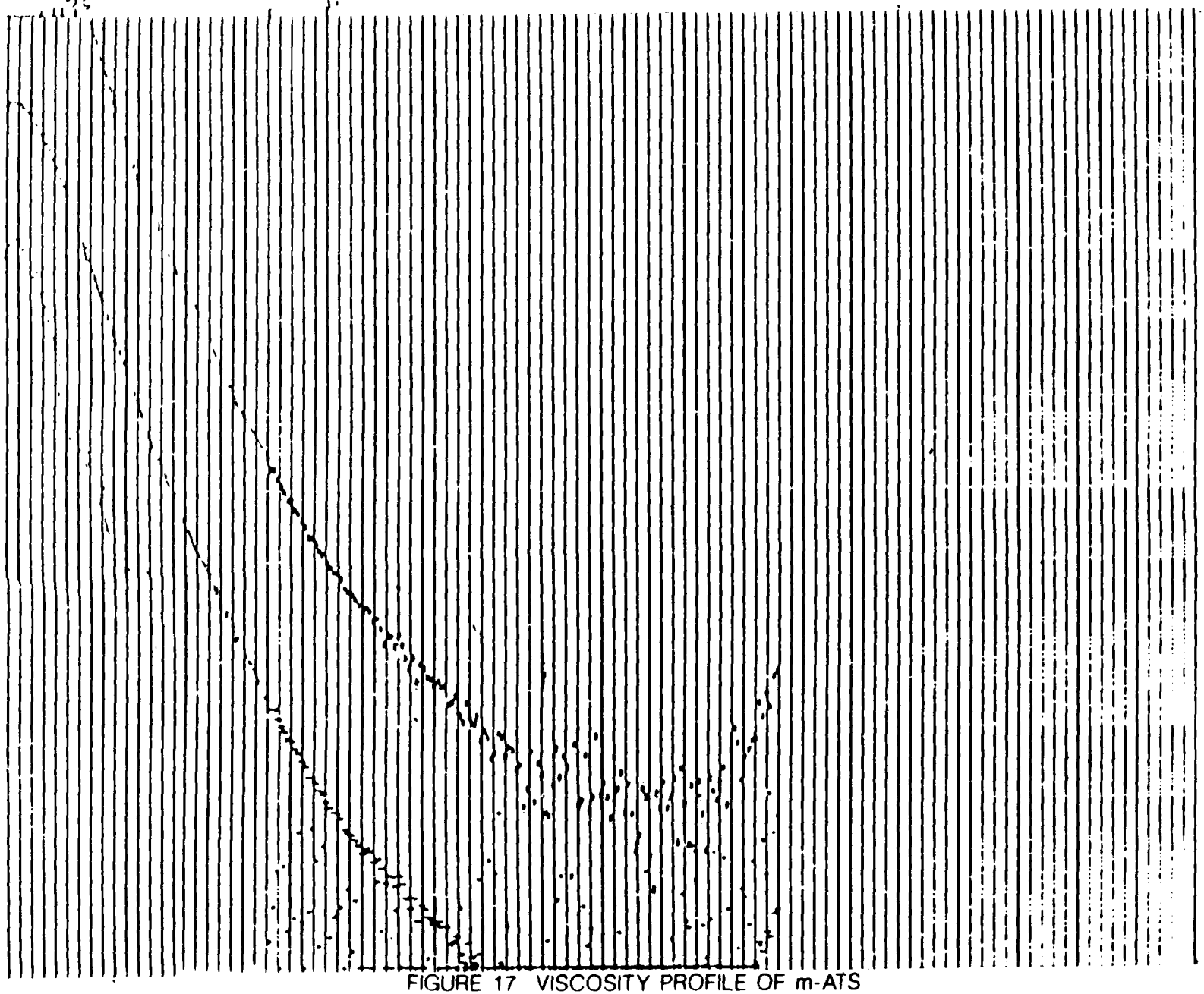
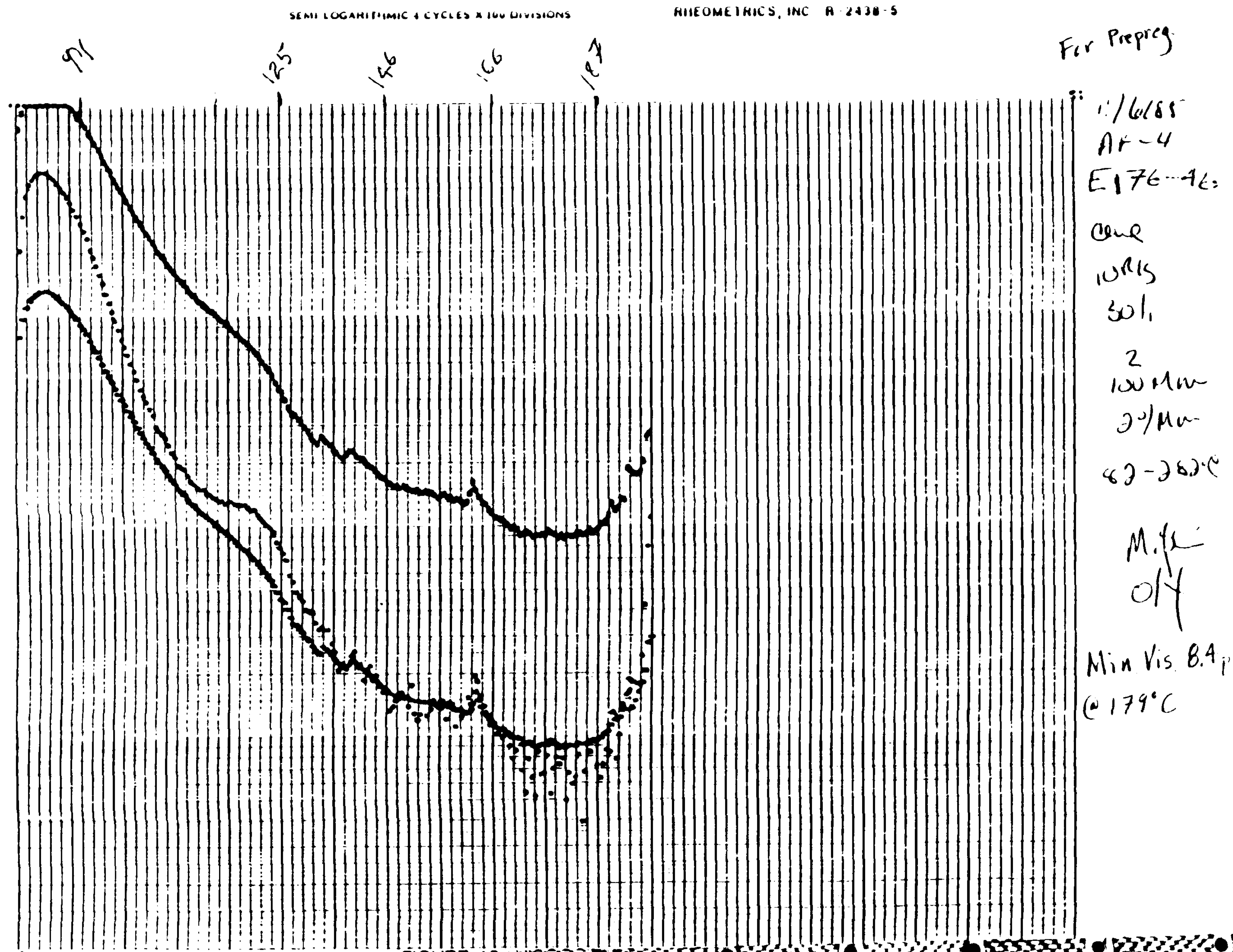


FIGURE 17 VISCOSITY PROFILE OF m-ATS


SEMI LOGARITHMIC 4 CYCLES X 100 DIVISIONS
RHEOMETRICS, INC R-2438-5
For Prepreg
91
125
146
166
187
1/6/85
Ar-4
E176-46
Cure
10RIS
30%
2
100 Mm
2°/Mm
60-280°C
Min Vis 8.4p
@ 179°C

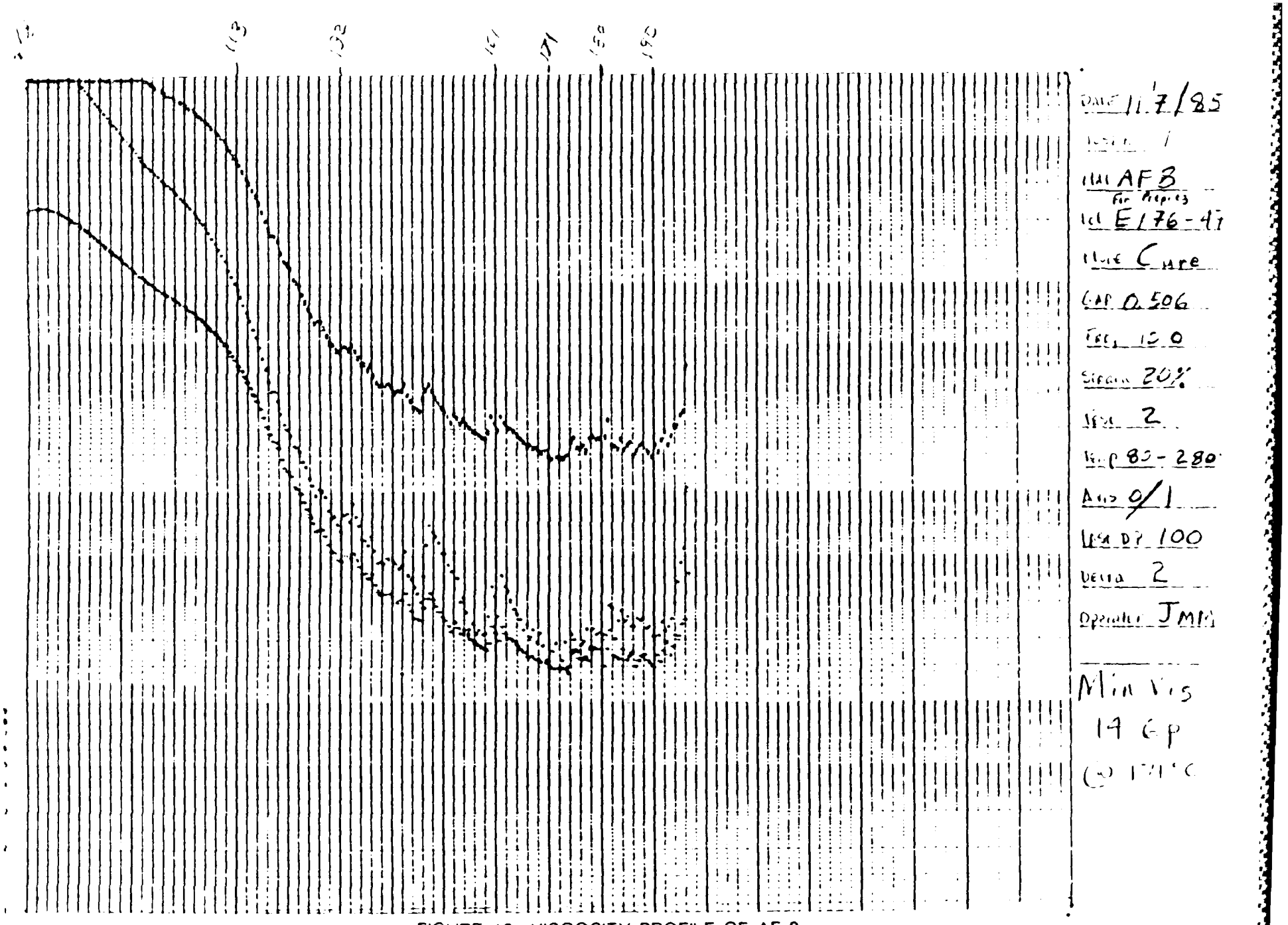


FIGURE 19 VISCOSITY PROFILE OF AF-8

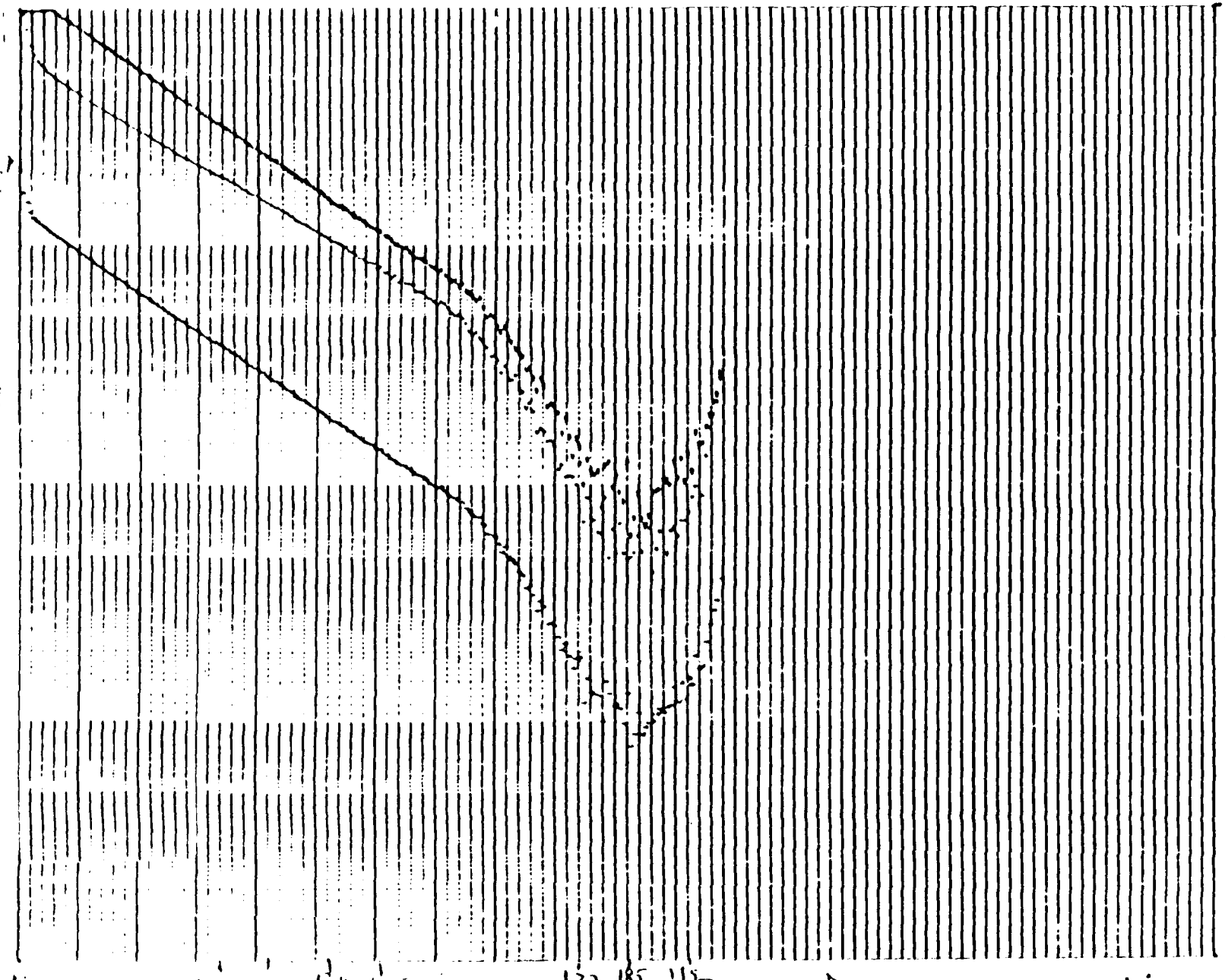


FIGURE 20. VISCOSITY PROFILE OF AF 11

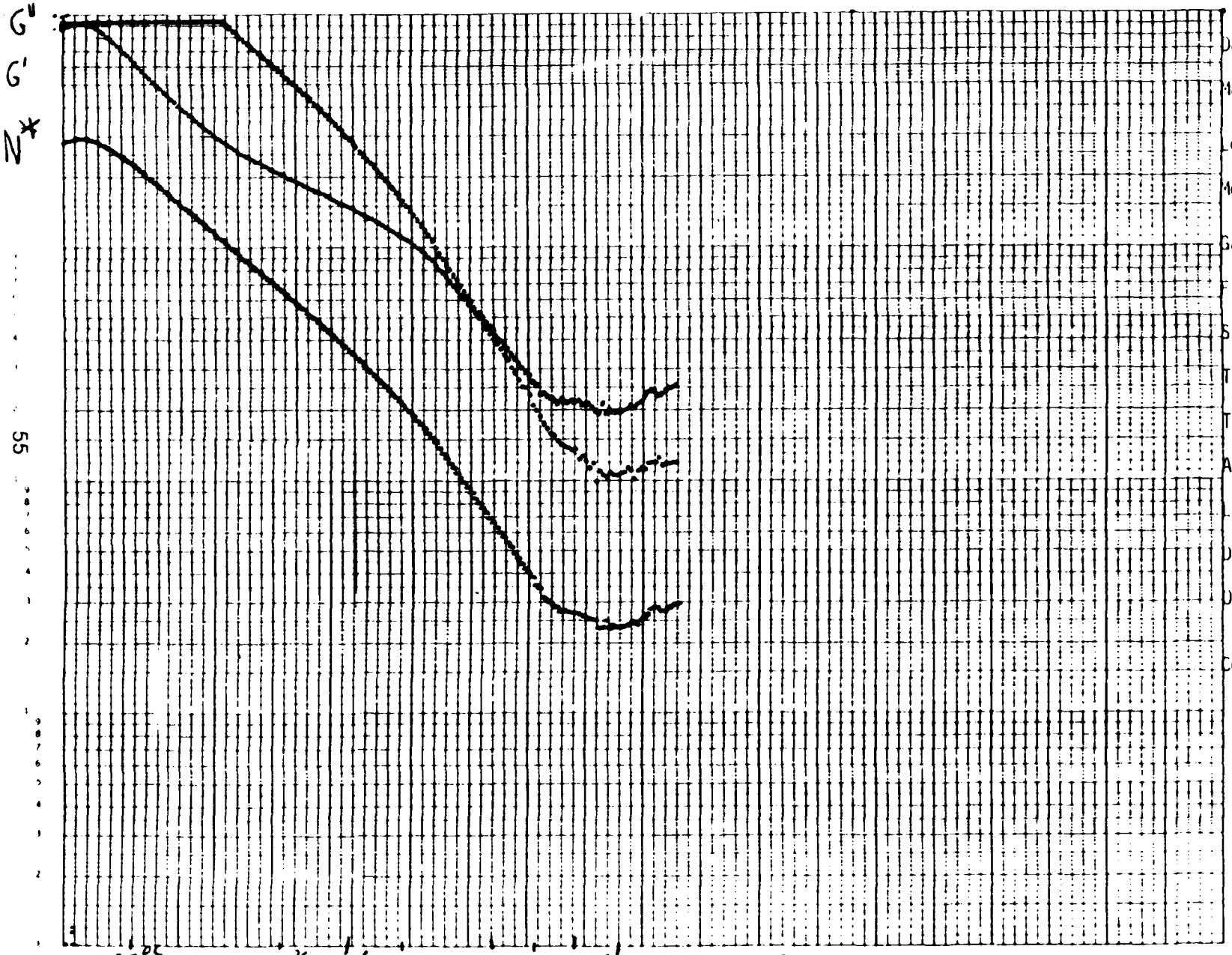


FIGURE 21 VISCOSITY PROFILE OF AF-20

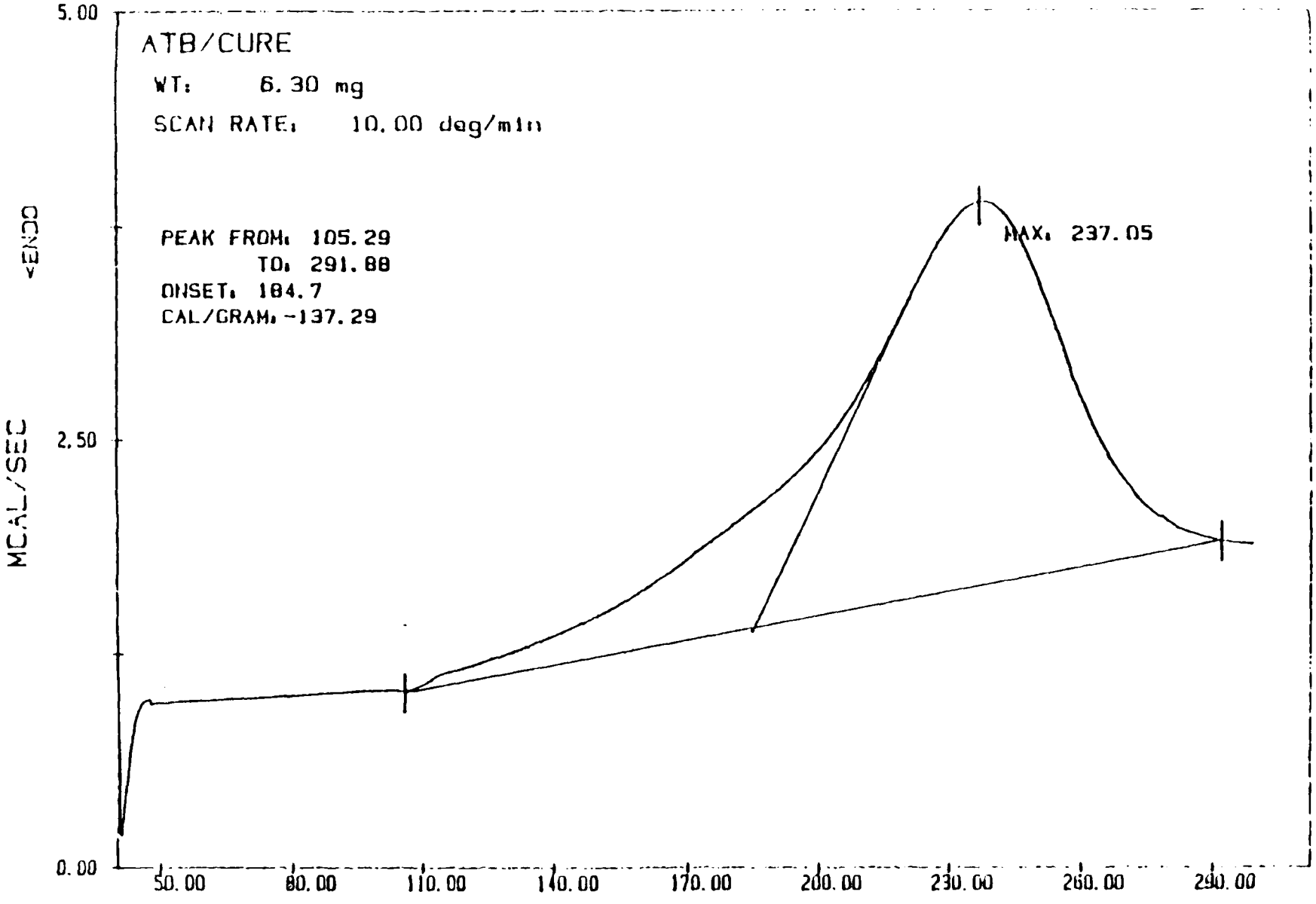


FIGURE 22 DSC OF m-ATB

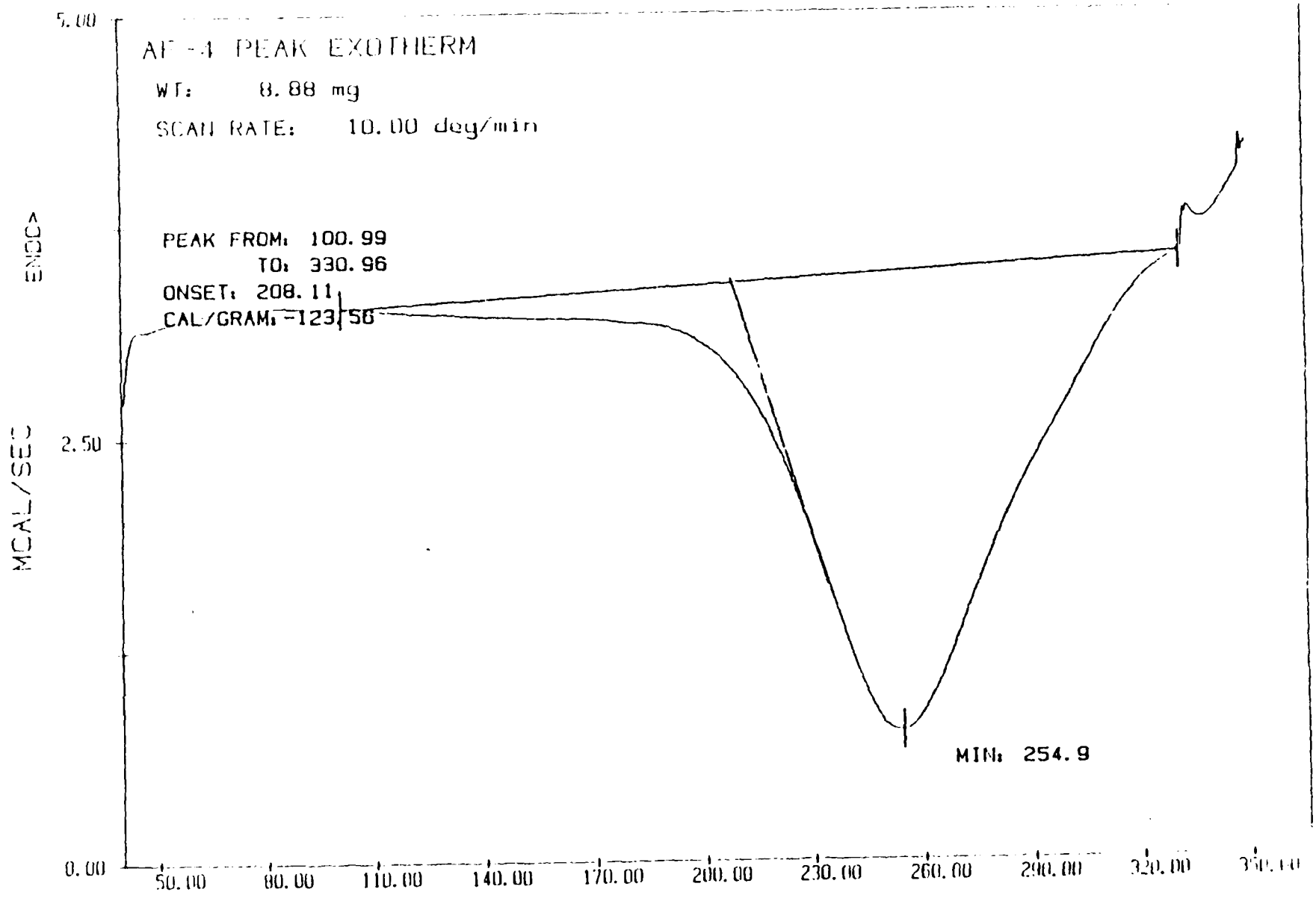


FIGURE 23 DSC OF AF-4

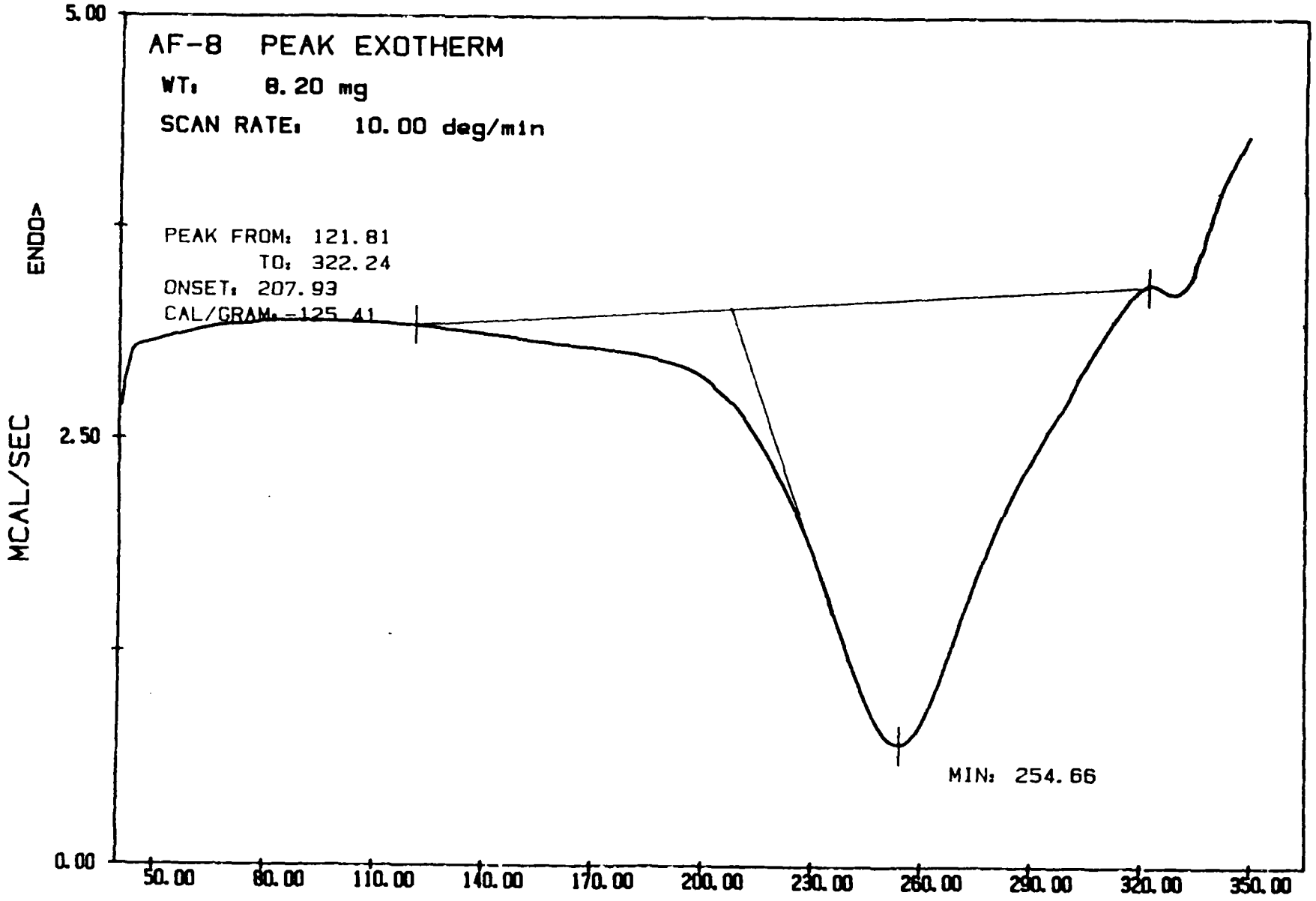


FIGURE 24 DSC OF AF-8

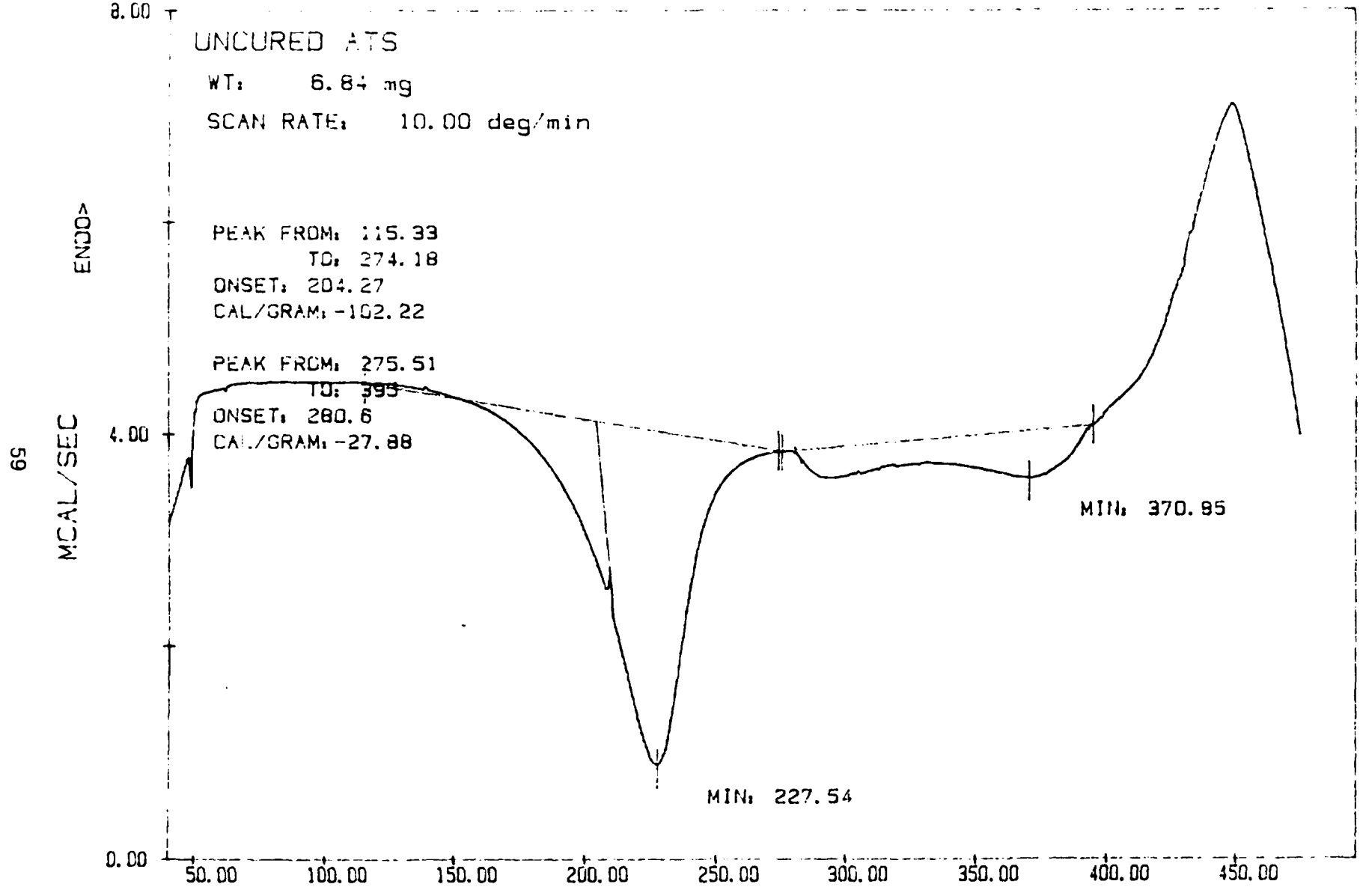


FIGURE 25 DSC OF m-ATS

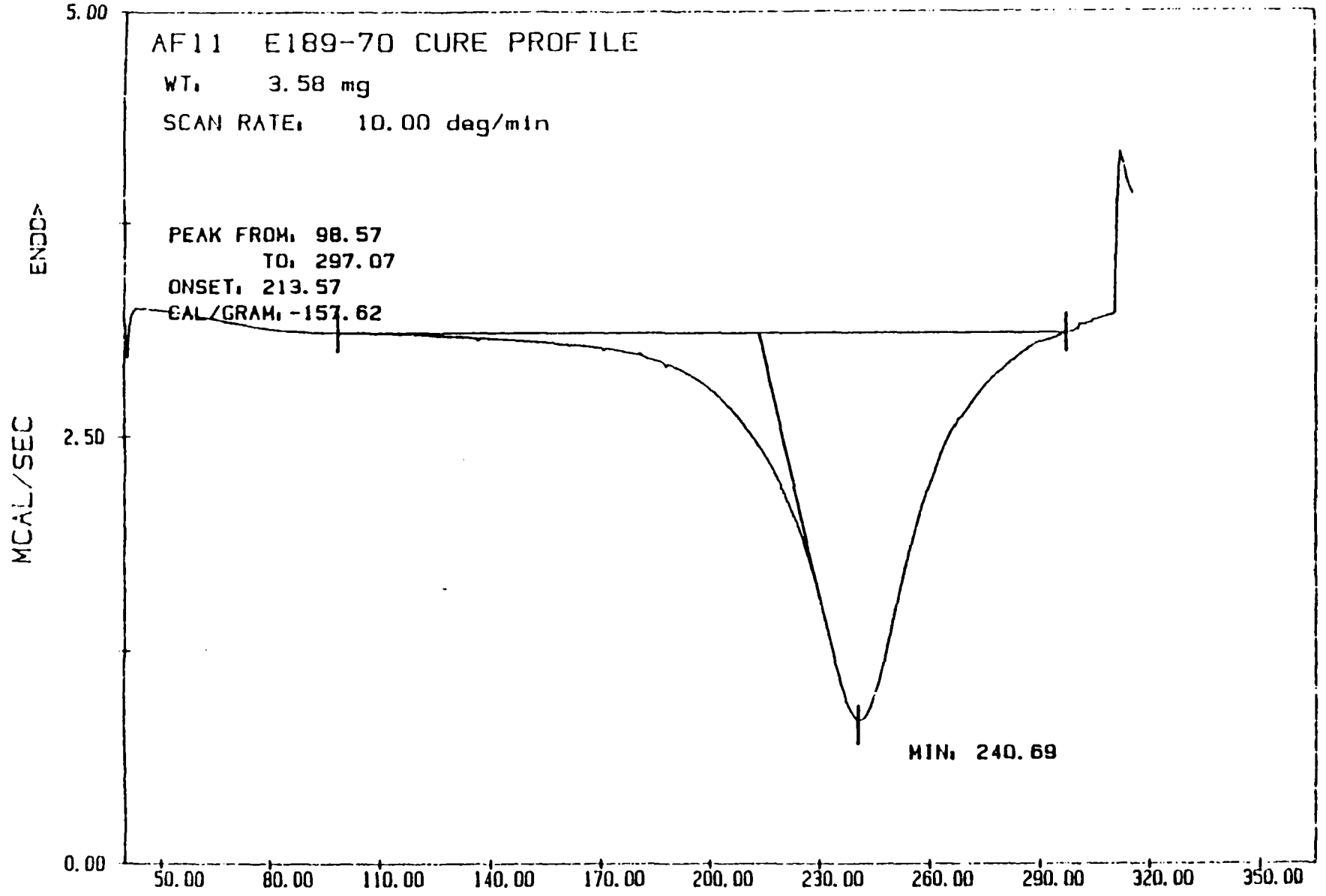


FIGURE 26 DSC OF AF-11

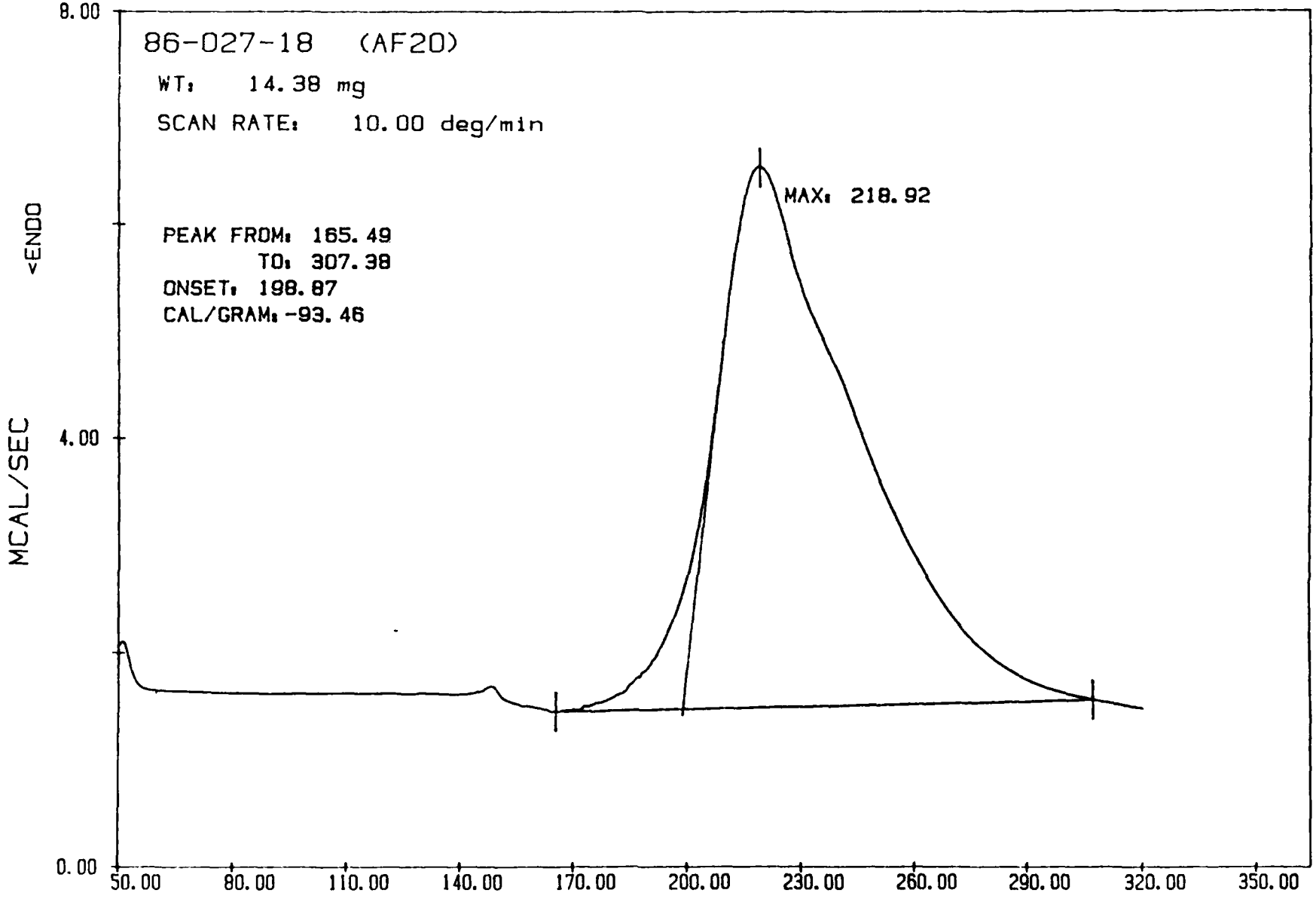
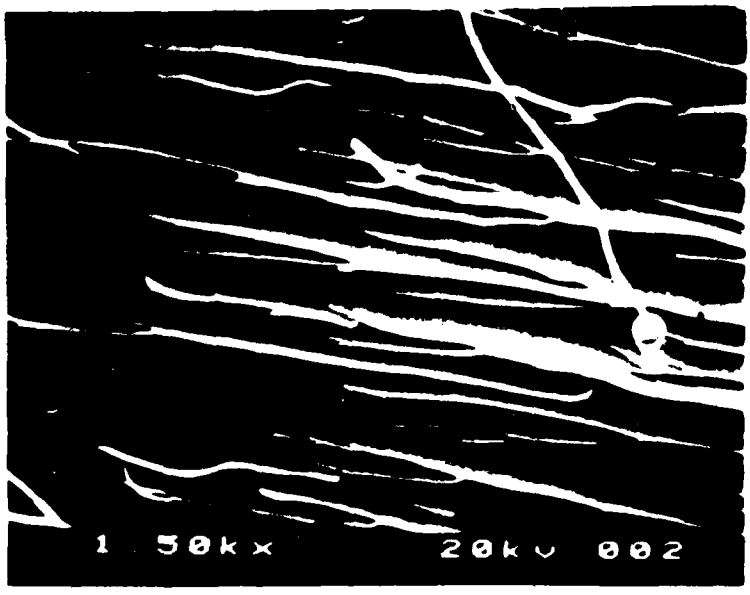
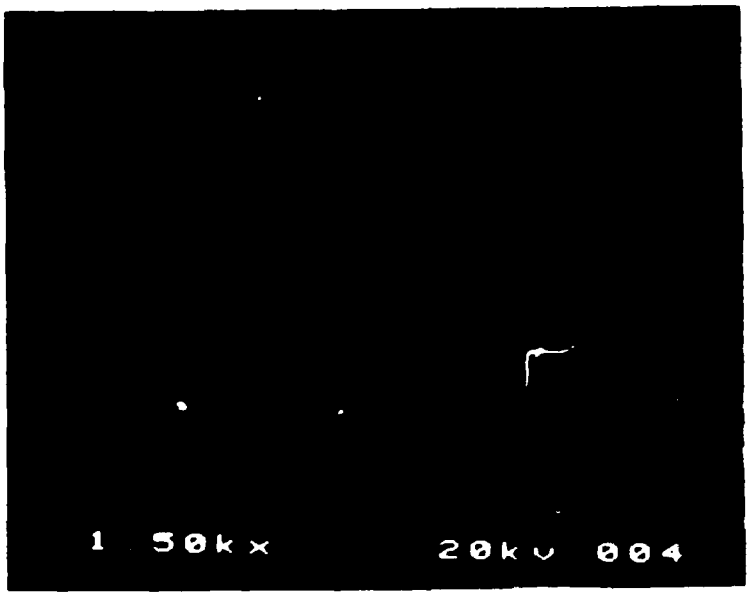


FIGURE 27 DSC OF AF-20



SEM PHOTOMICROGRAPH OF CURED m-ATB



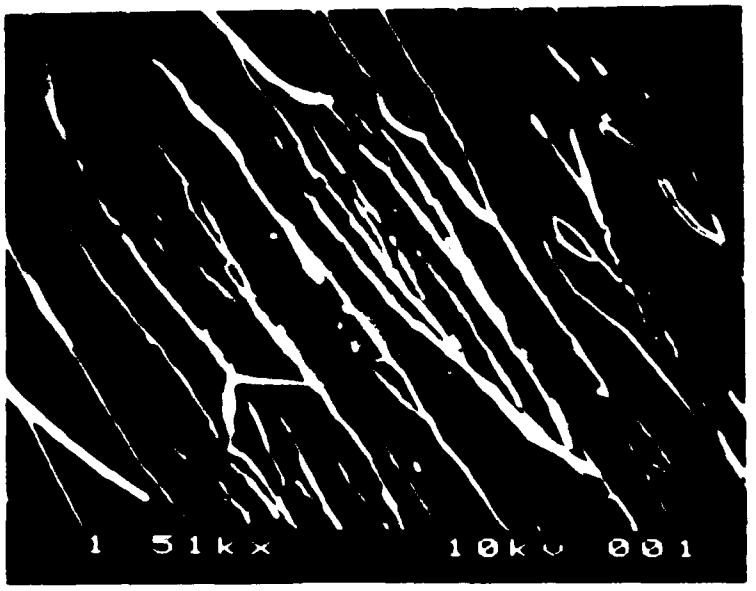
SEM PHOTOMICROGRAPH OF CURED m-ATS

FIGURE 28

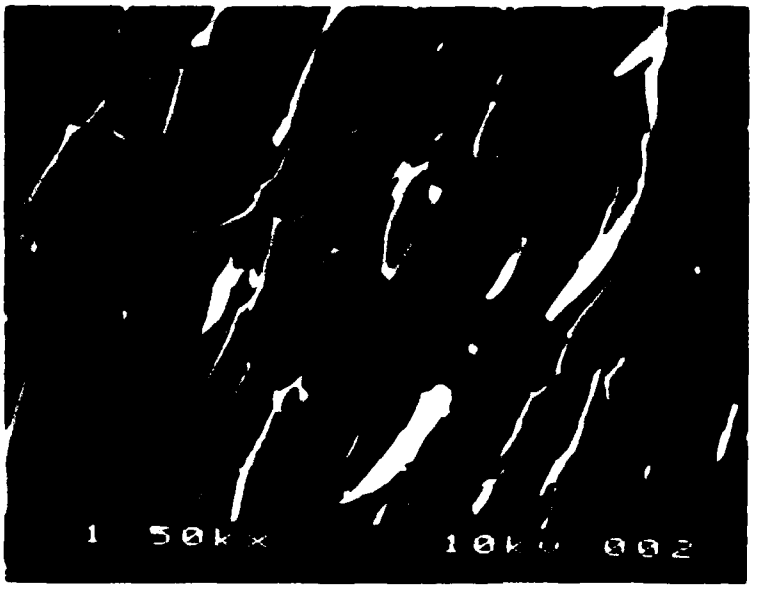
SEM PHOTOMICROGRAPH OF CURED AF-8

SEM PHOTOMICROGRAPH OF CURED AF-4

FIGURE 29



SEM PHOTOMICROGRAPH OF CURED AF-20



SEM PHOTOMICOGRAPH OF CURED AF-11

FIGURE 30

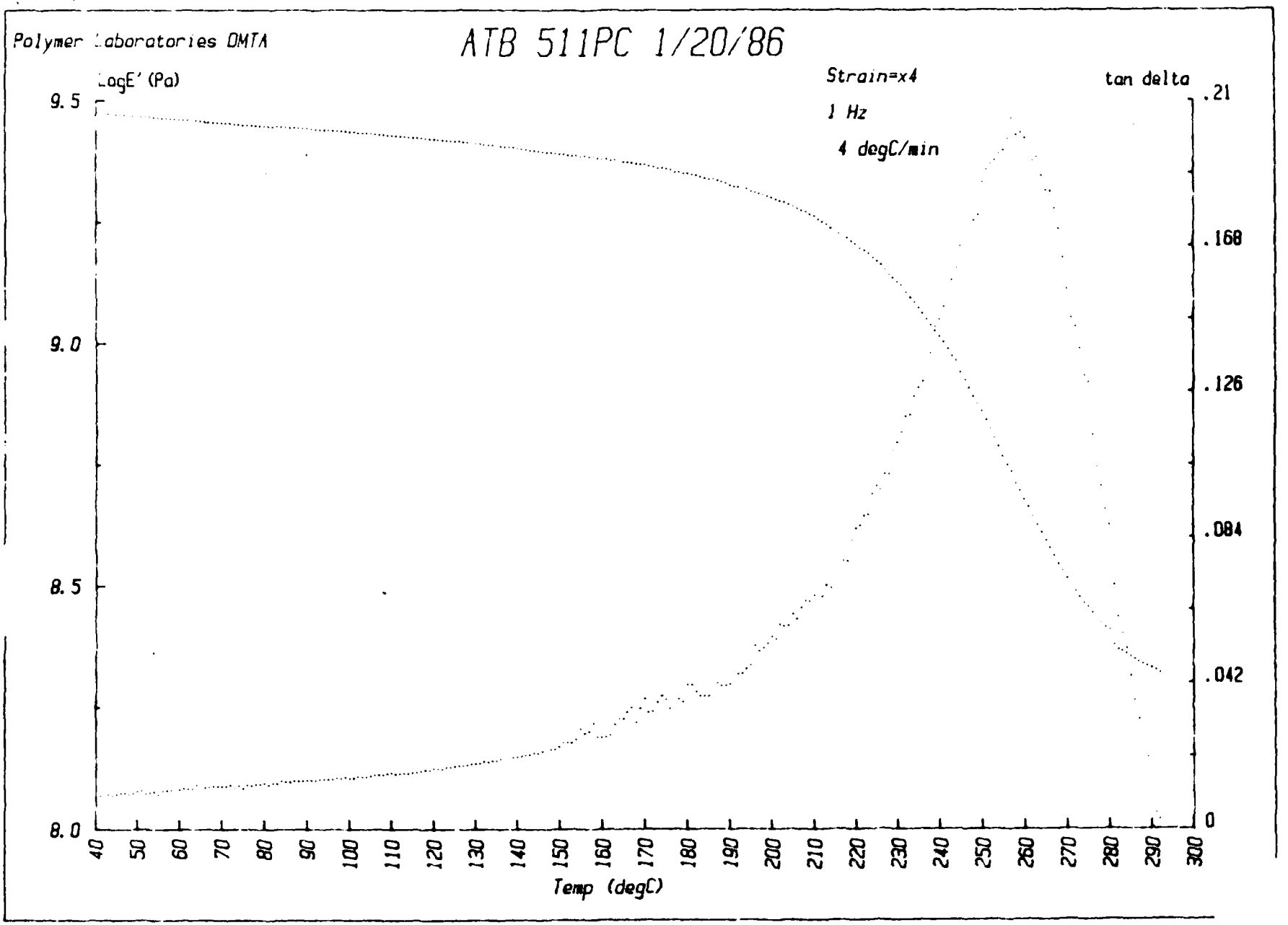


FIGURE 31 DMTA OF m-ATB

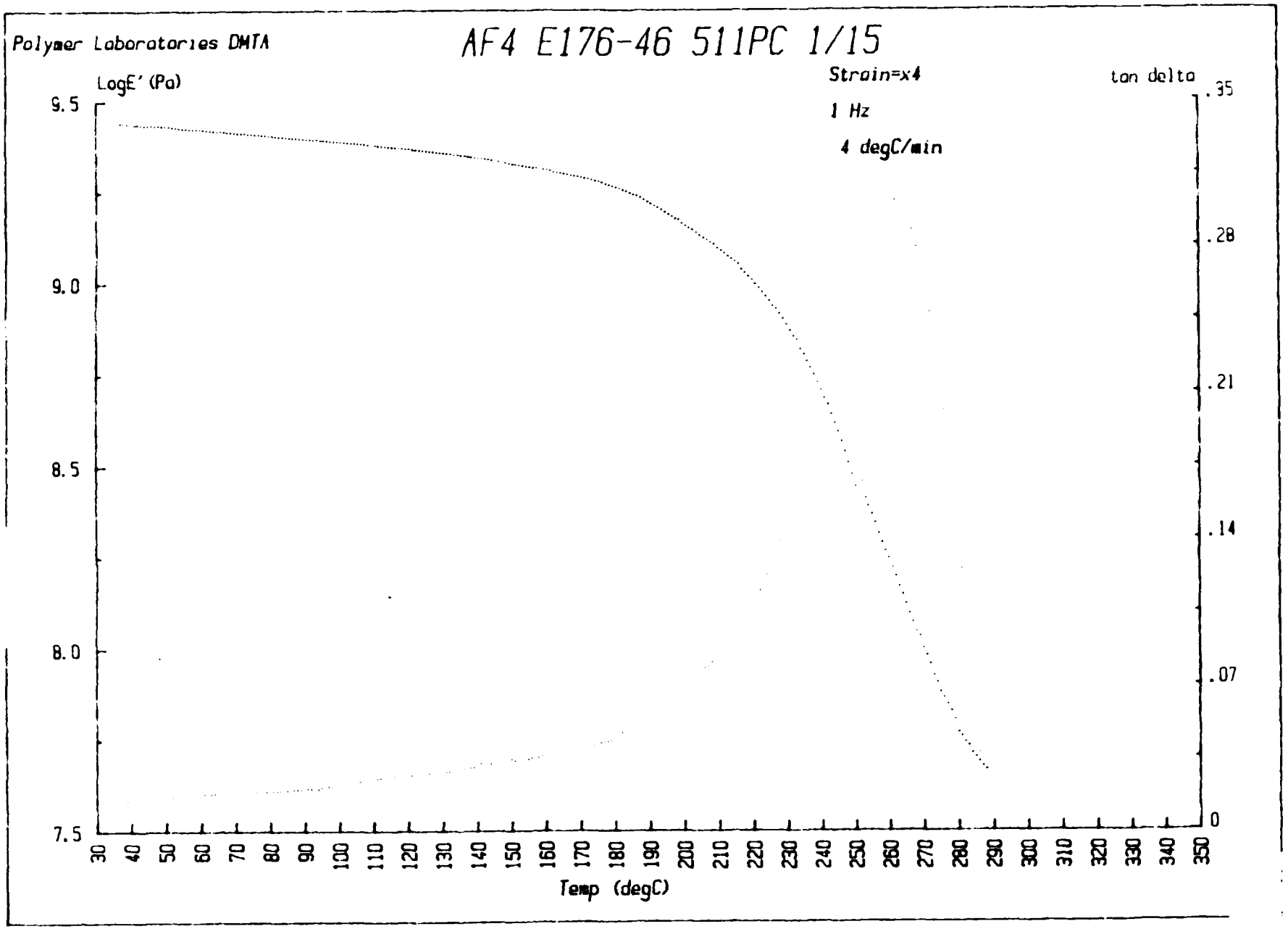


FIGURE 32 DMTA OF AF-4

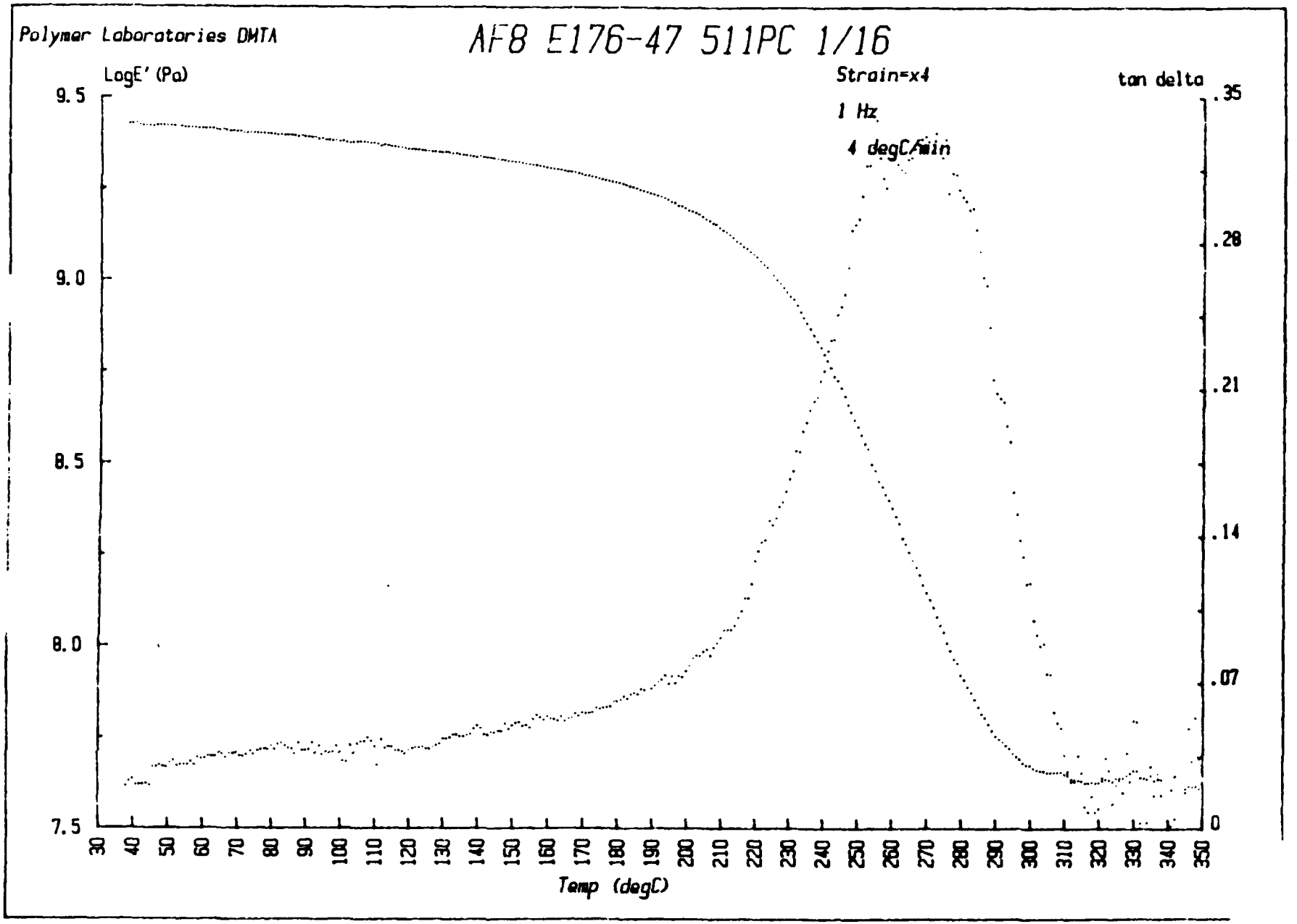


FIGURE 33 DMTA OF AF-8

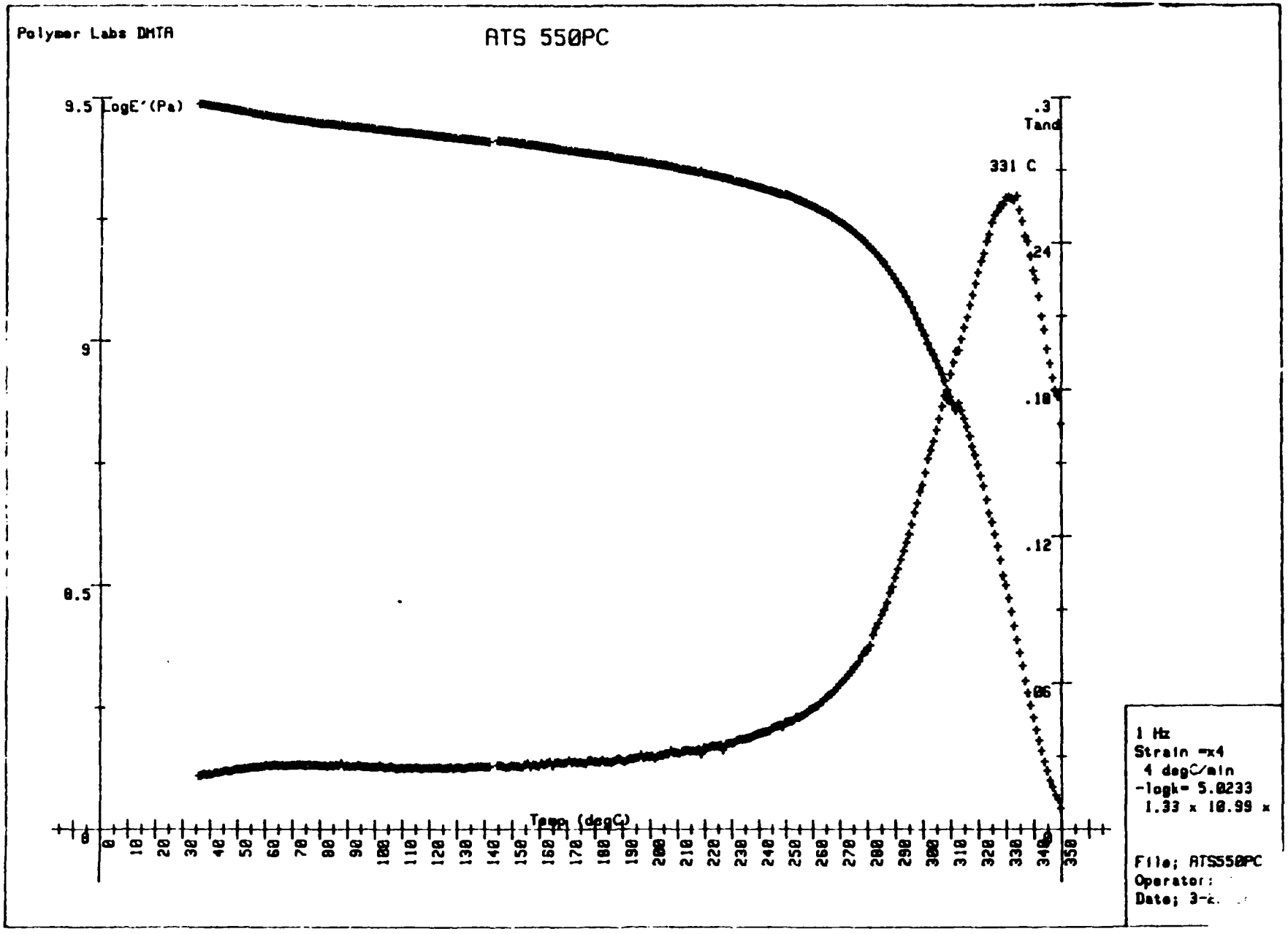


FIGURE 34 DMTA OF m-ATS

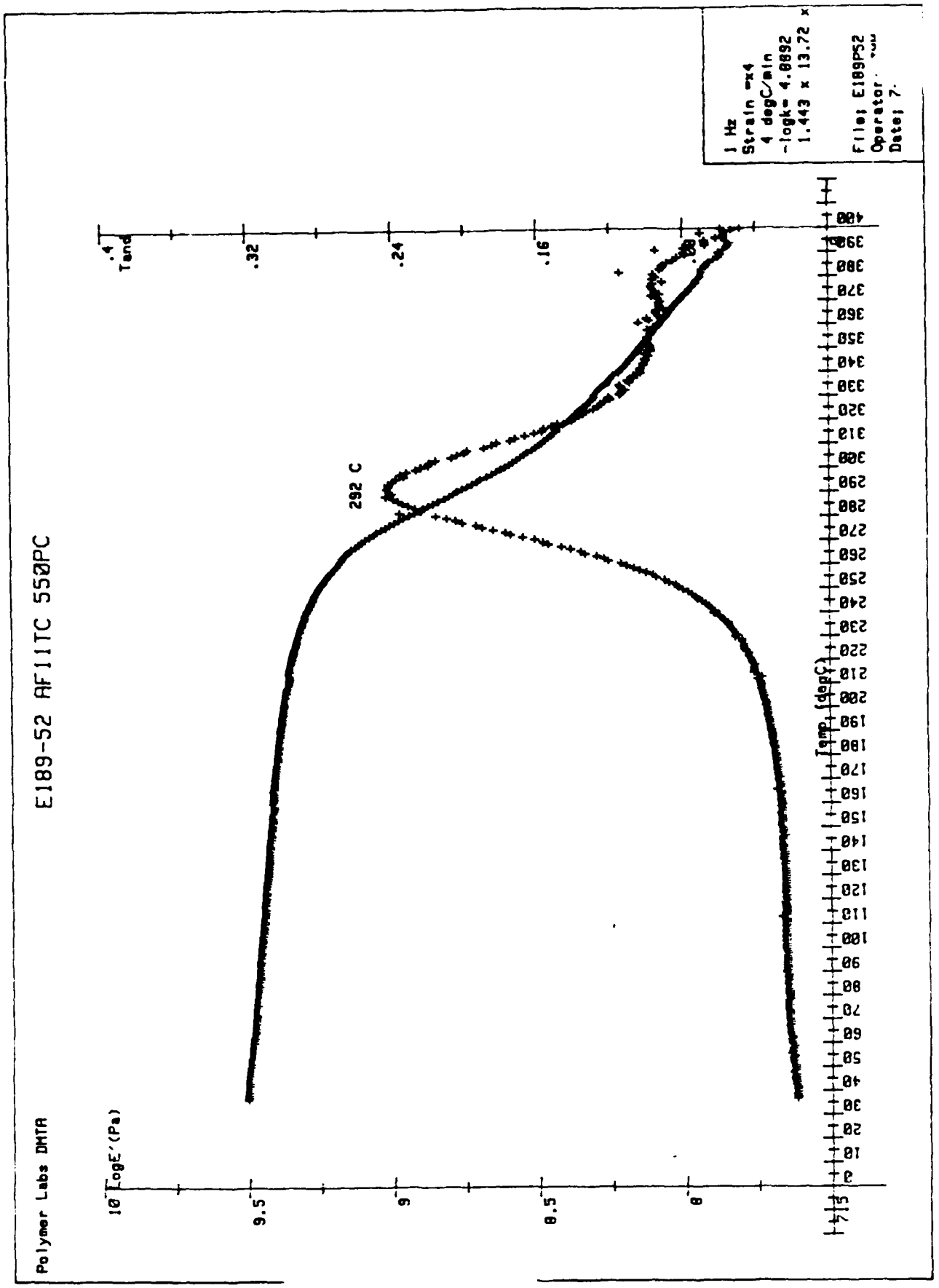


FIGURE 35 DMTA OF AF-11

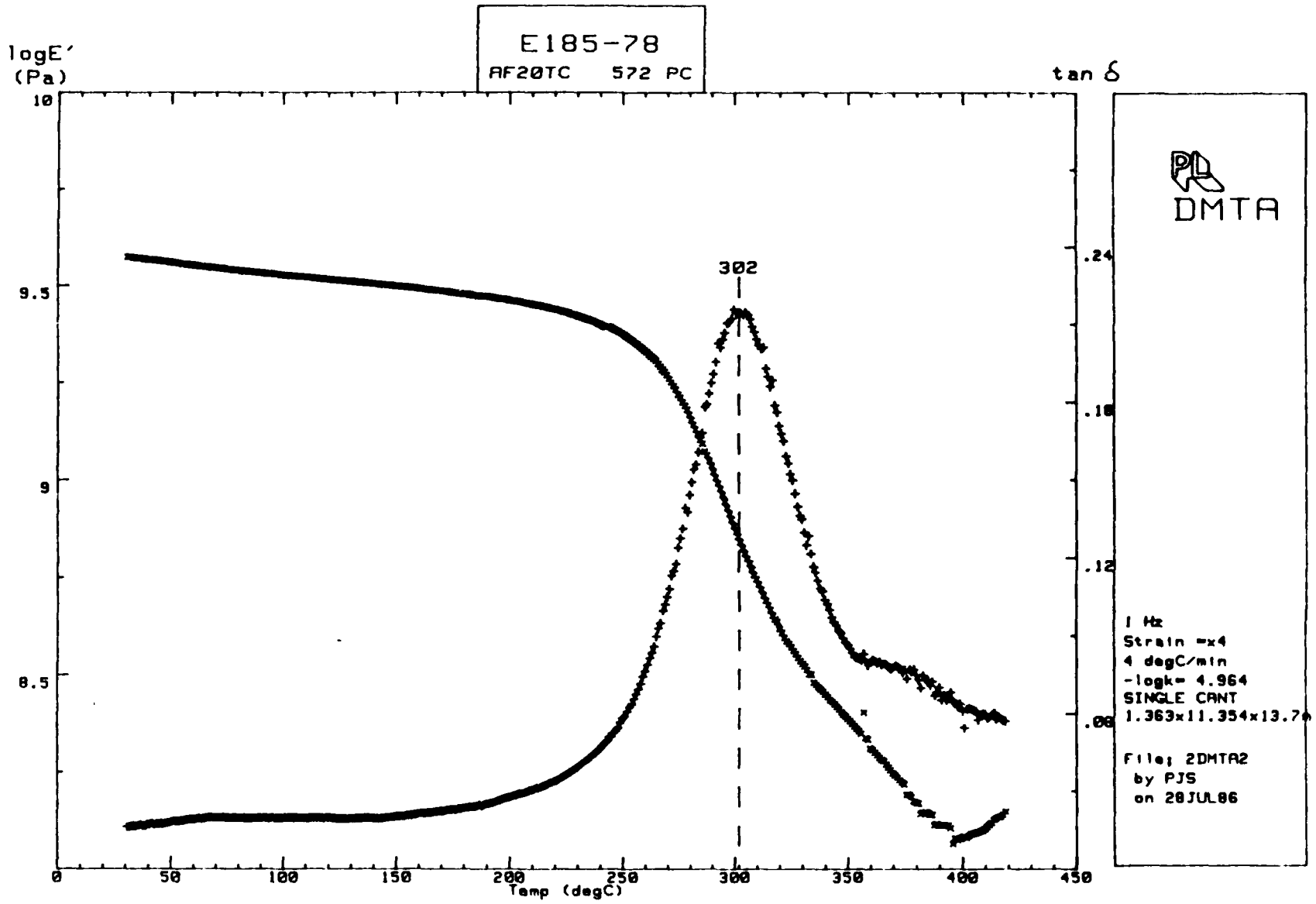


FIGURE 36 DMTA OF AF-20

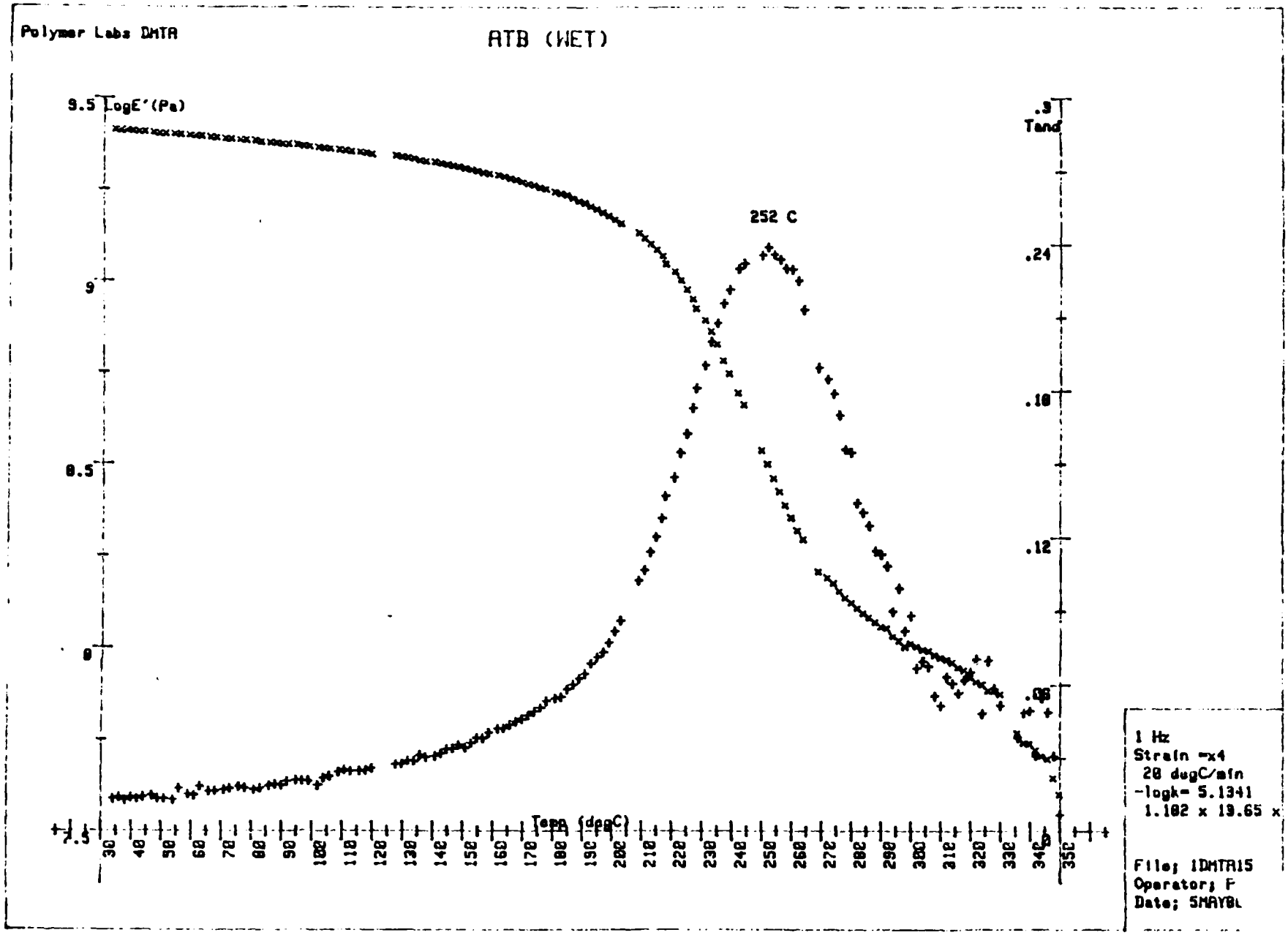


FIGURE 37 WET DMTA OF m-ATB

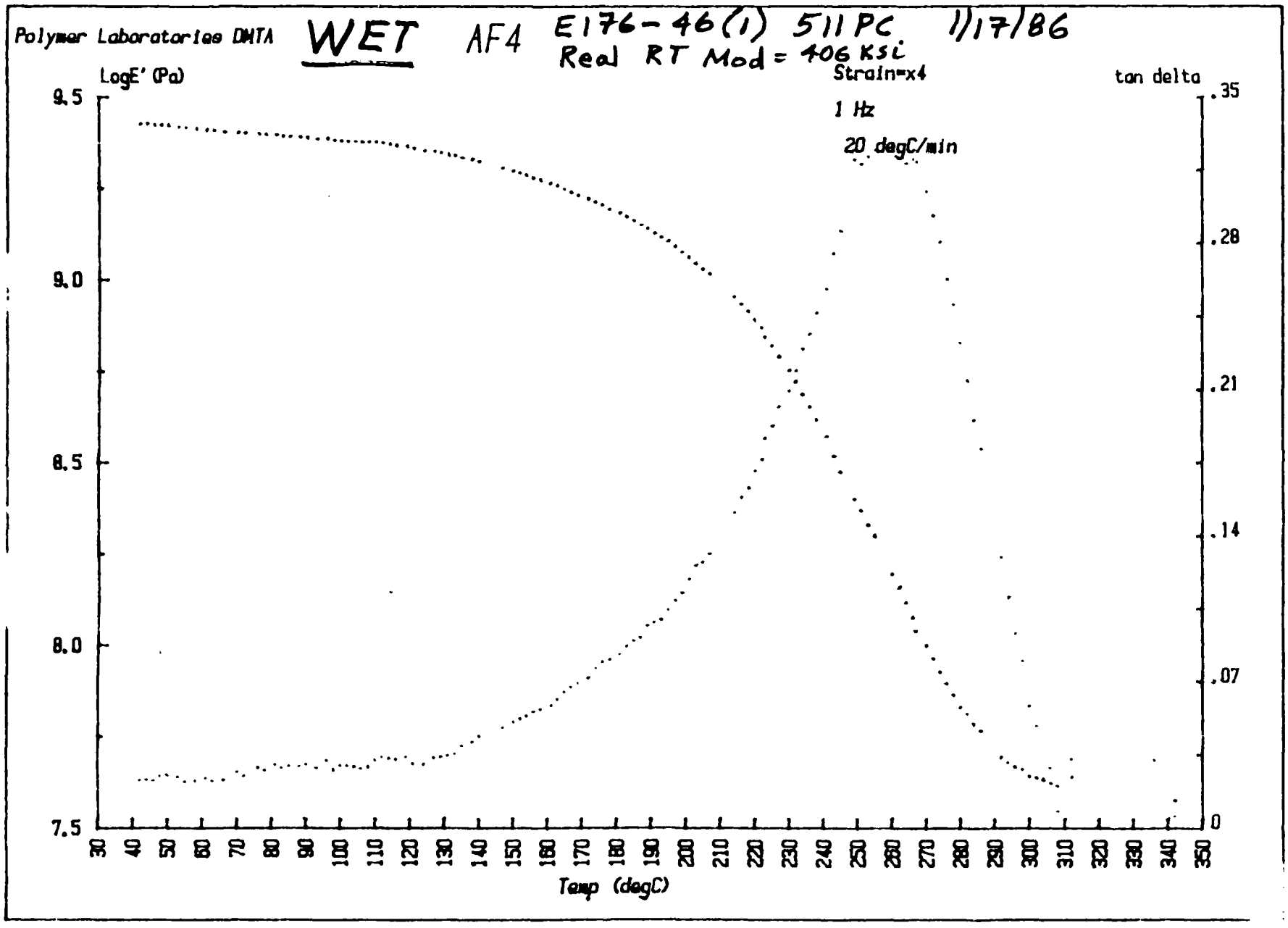


FIGURE 38 WET DMTA OF AF-4

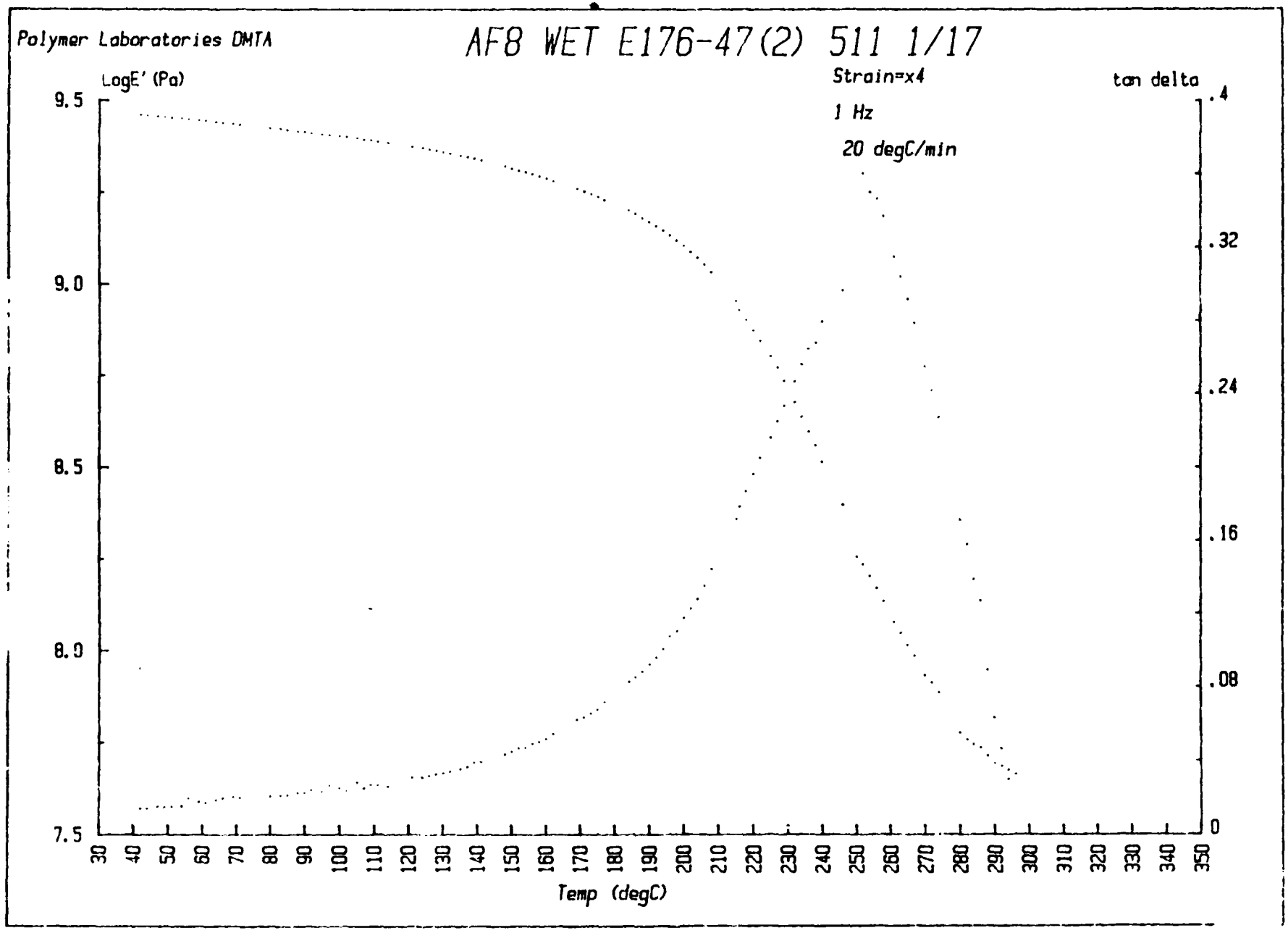


FIGURE 39 WET DMTA OF AF-8

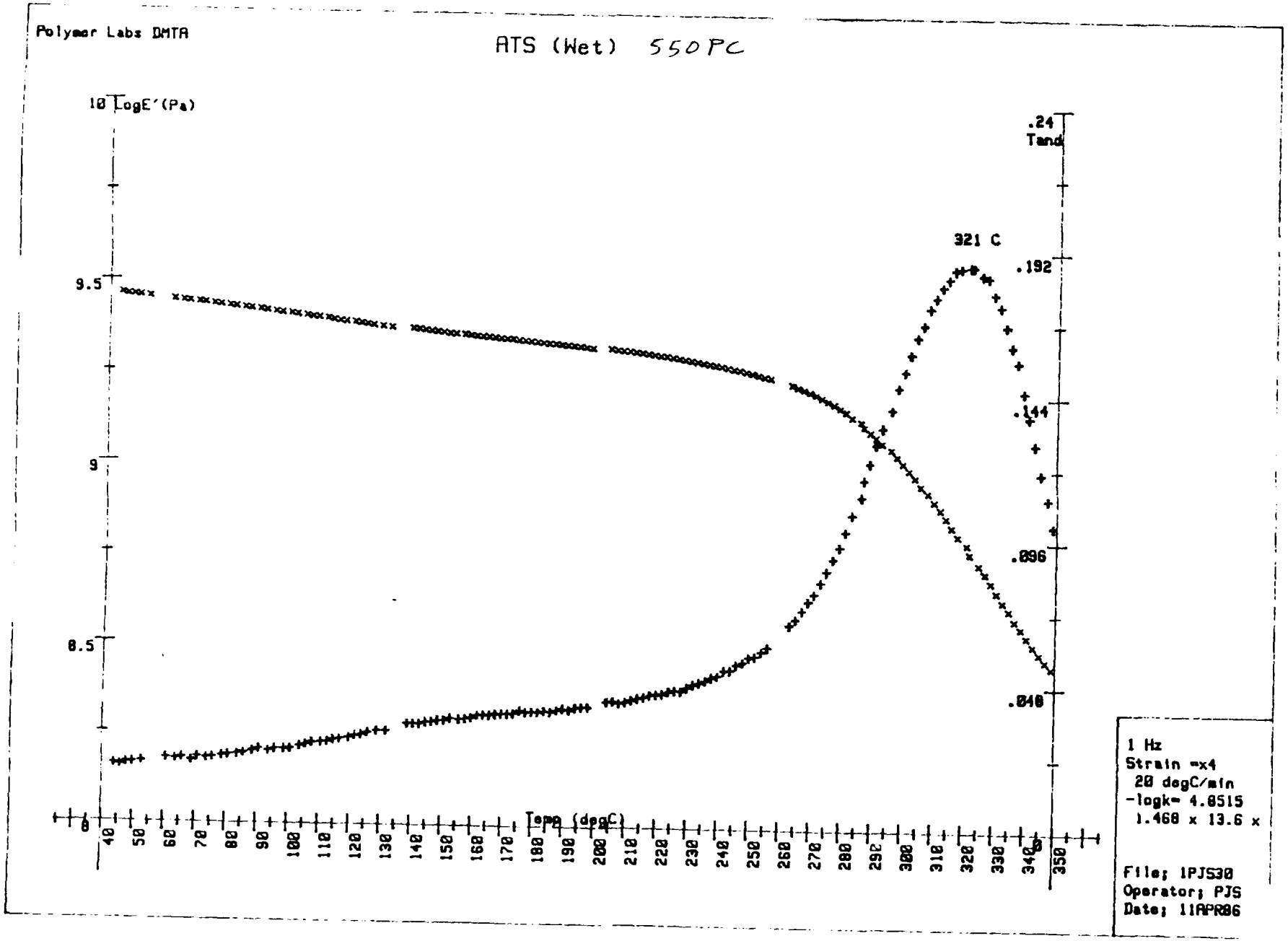


FIGURE 40 WET DMTA OF m-ATS

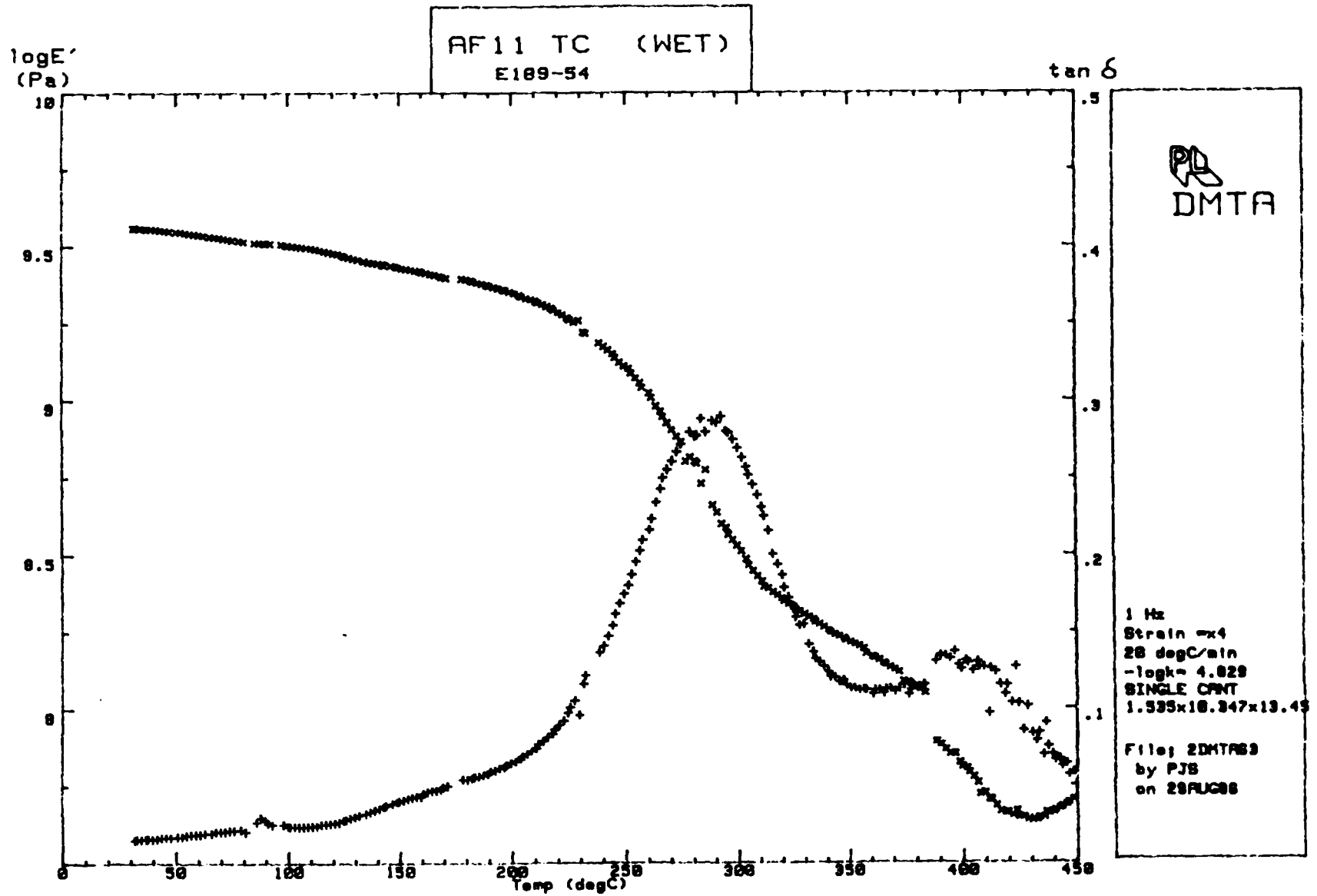


FIGURE 41 WET DMTA OF AF-11

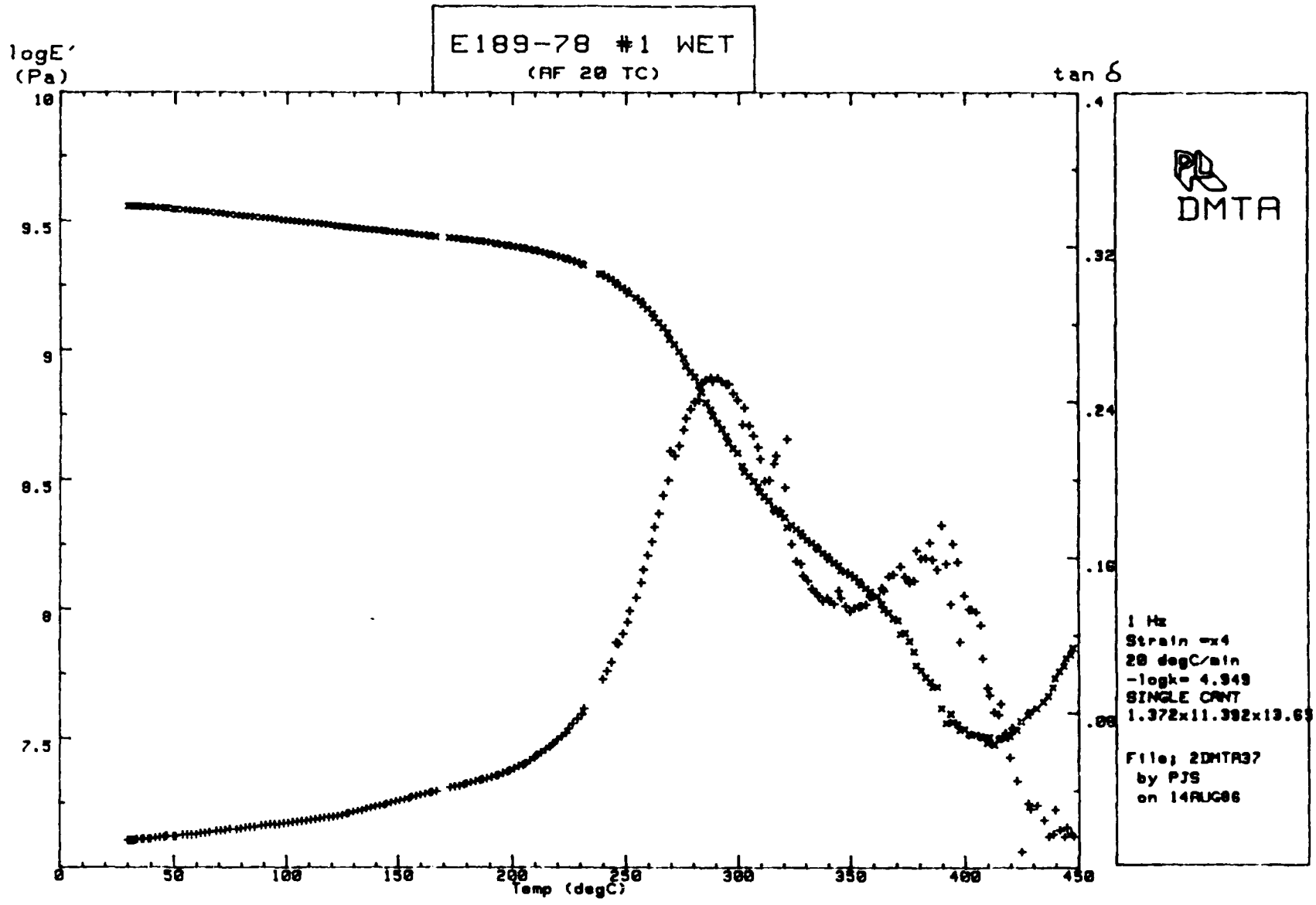


FIGURE 42 WET DMTA OF AF-20

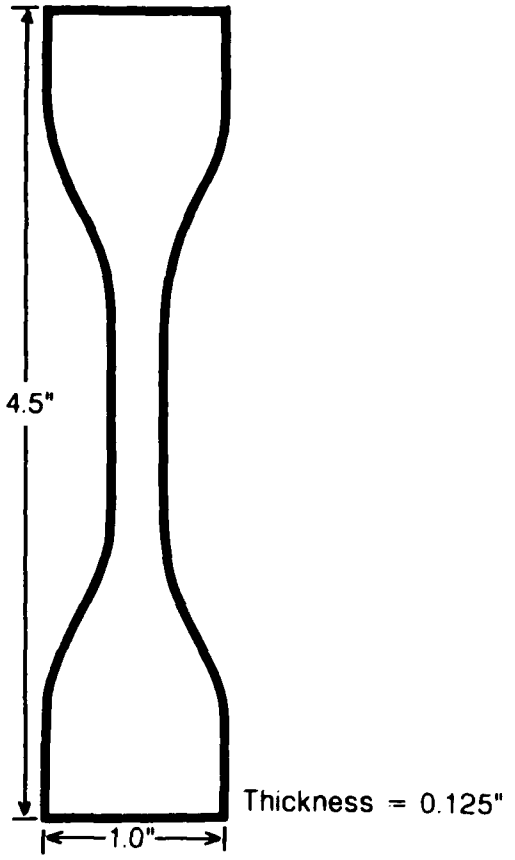


FIGURE 43 NEAT RESIN TENSILE DOGBONE

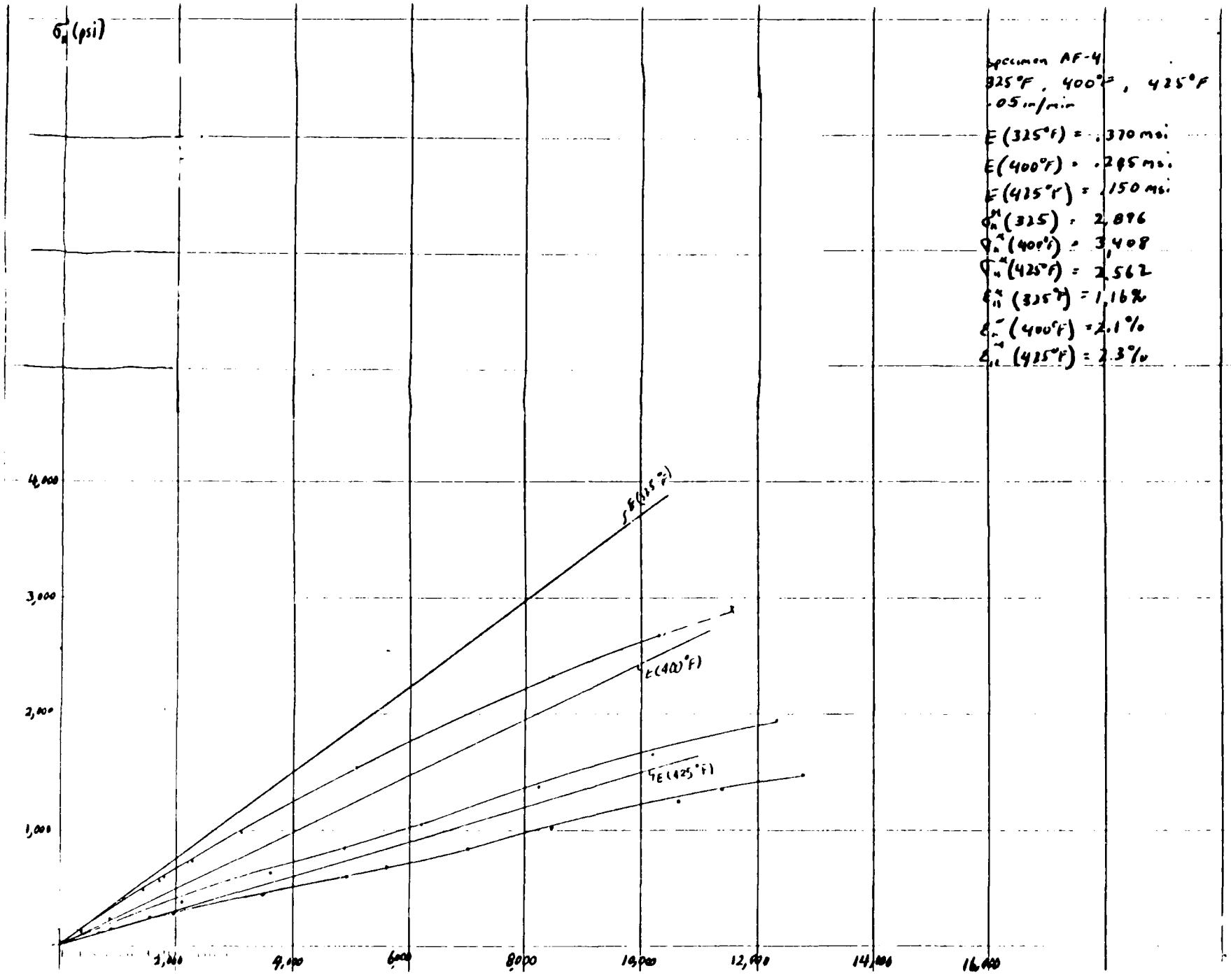


FIGURE 44 ELEVATED TEMPERATURE AF-4 RESIN DOGBONE STRESS-STRAIN CURVES

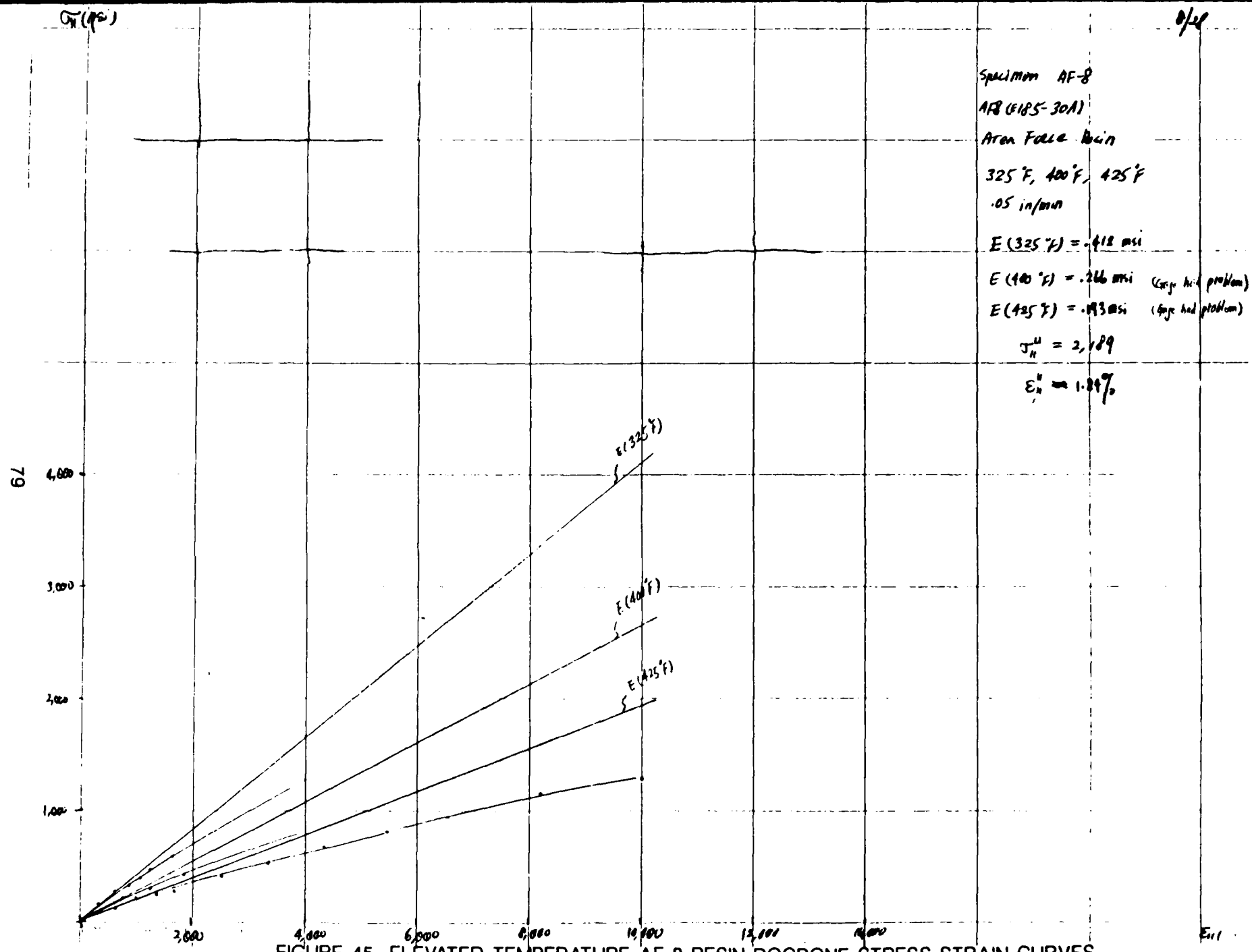


FIGURE 45 ELEVATED TEMPERATURE AF-8 RESIN DOGBONE STRESS-STRAIN CURVES

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